HIGHWAY RESEARCH **RECORD**

Number
315

Soil Stabilization:
Multiple Aspects

11 Reports

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Contents

Concepts for Developing Stabilization Recommendations for the Soils of an Area

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•THERE IS AN INCREASING DEMAND for quality paving materials at all levels of the highway system (local, state, and federal). These quality materials must economically provide desirable strength and durability characteristics. In certain areas of Illinois as well as in many other areas of the United States, quality natural materials meeting these requirements are not locally available and must be transported a large distance at considerable cost. It is apparent that, from the economic standpoint, it is desirable to utilize locally available materials to the greatest extent possible. In many instances, quality paving materials can be obtained by appropriately modifying or stabilizing local and on-site materials.

Although considerable work has been done in the field of material beneficiation (modification and stabilization), suitable criteria are not generally available for determining which procedures or techniques can feasibly be used with a particular material in order to achieve maximum benefits at minimum cost. This is particularly true with natural soils or unprocessed granular materials.

This paper outlines the pertinent aspects of a research project that was aimed at developing stabilization recommendations for the surficial soils of Illinois.

RESEARCH OBJECTIVE

The general objective of the investigation was concerned with maximum utilization of local and on-site materials in pavement construction. Specific objectives were as follows:

1. Develop, for typical Illinois surficial deposits, guidelines and criteria necessary to determine the applicability of current materials beneficiation techniques; and

2. Develop, for specific Illinois surficial soils and materials, feasibility recommendations that will provide for effective and economical utilization of various stabilization techniques and promote maximum utilization of local materials.

This investigation was limited to a study of the applicability and utilization of the more common beneficiation techniques (lime, cement, bitumen, and lime-fly ash) and the exploration of a limited number of new techniques (combination stabilization and blending).

DEVELOPMENT OF TENTATIVE STABILIZATION GUIDELINES AND CRITERIA

Effective and economical application of various stabilization techniques can be obtained only if the capabilities and limitations of stabilizing agents are well established and understood. A literature review (1) was conducted to establish the type and nature of the stabilization reactions afforded by the various stabilizers and to establish a soil's or an aggregate's chemical and physical characteristics that affect the ability of a particular stabilizing agent to properly modify or stabilize the soil or the aggregate.

Two broad categories of stabilizing agents exist with respect to the stabilization mechanisms effected when the agent is mixed with water and a soil or aggregate. "Active" stabilizing agents, a prime example of which is lime, cause chemical reactions

to occur in the soil-water-stabilizer system. These reactions are responsible for the improved engineering characteristics of the treated mixture. With this type of stabilizing agent, the chemical properties of a soil are very important. The literature indicated that soil properties such as organic matter content, natural soil pH, degree of weathering, type of internal drainage, horizon in the pedologic profile, predominant type of clay mineral, and, to a certain extent, texture and plasticity greatly affect the modifying or stabilizing ability of the active stabilizing agents.

"Inert" stabilizing agents, an example of which is bituminous materials, do not react chemically with the soil or aggregate, but rather provide modification or stabilization to the system by increasing the cohesion or water-proofing or both of the treated mixture. With this type of stabilizing agent, physical characteristics of the soil such as gradation, texture, and plasticity are the predominant factors that control the modifying or stabilizing ability.

Many other stabilizing agents such as cement and lime-fly ash display both active and inert characteristics, and thus both chemical and physical soil properties are important.

With the information derived from the literature survey, it was possible to establish tentative guidelines and criteria that could be used to promote effective utilization of different beneficiation techniques.

VERIFICATION OF THE APPLICABILITY OF THE TENTATIVE STABILIZATION GUIDELINES AND CRITERIA TO ILLINOIS SOILS AND MATERIALS

The next step after establishing the tentative stabilization guidelines and criteria was to verify their applicability to Illinois soils and materials. To verify the applicability of the tentative guidelines and criteria, it was necessary to sample representative Illinois surficial materials and to determine their response to treatment with various beneficiation techniques.

In order to plan an appropriate sampling program, the nature and distribution of the surficial deposits of Illinois were established. Relevant publications from the Agricultural Experiment Station, Illinois Geological Survey, and various other sources of published and unpublished information were reviewed, and a short summary was prepared (2).

Soils of Illinois

The surficial soils of Illinois are mainly derived from loess and relatively young Wisconsin age glacial drift, but in some areas older more highly weathered Illinoian age glacial drift may be found close to the surface. Figure 1 shows the extent and distribution of the main parent materials found in Illinois (8).

The surficial deposits of Illinois can be placed into 3 general engineering groupings: (a) highly plastic, high-clay content, fine-grained A-6 and A-7 AASHO classified soils; (b) medium-textured A-4 and low-plasticity A-6 soils; and (c) coarse-textured A-1, A-2, and A-3 gravelly and sandy soils. Data given in reference 5 indicate that a substantial proportion of the parent materials (material below the zone of weathering) of Illinois are medium-textured A-4 and low-plasticity A-6 soils. However, in general, unweathered

LOESS

OUTWASH

GLACIAL TILL

ALLUVIUM

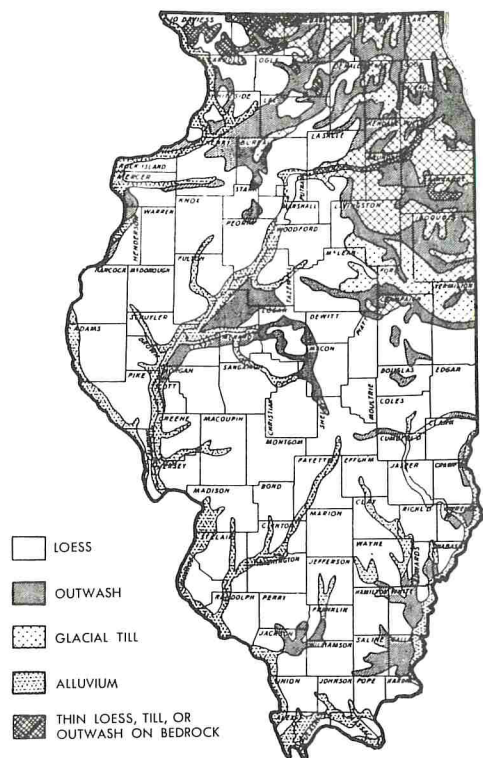THIN LOESS, TILL, OR OUTWASH ON BEDROCK

Figure 1. The extent of the soil parent materials in Illinois.

TABLE 1
SUMMARY OF CHARACTERISTICS OF SOIL SAMPLES

Sample	Location	Description	AASHO	LL	PI	Percent < 2μ Clay
Wisconsin loam till	Champaign County	Calcareous loam till of Wisconsin age	A-4(6)	23	7	16
Illinoian till	Sangamon County	Calcareous loam till of Illinoian age	A-6(6)	25	11	14
Fayette B	Henry County	Profile developed in calcareous silt loam	A-7-6(12)	57	34	34
Fayette C	Henry County	Peorian loess	A-4(8)	32	10	21
Plainfield sand	Cass County	Outwash deposit in Illinois River bottom	A-3(0)		NP	2
Hamburg sandy loam	Cass County	Calcareous loess from Illinois River bluff	A-4(8)	26	2.5	9
Dickinson B	Whiteside County ⎫	Profile developed in	A-2-4(0)	20	2	8
Dickinson C	Whiteside County ⎬	sandy outwash material	A-2-4(0)		NP	8
Alvin B	Lawrence County ⎫	Profile developed in	A-4(4)	20	1	15
Alvin C	Lawrence County ⎬	sandy outwash material	A-4(4)		NP	13
Sandy loam till	McHenry County	Calcareous sandy loam till of Wisconsin age	A-1-b(0)		NP	4
Pit-run gravels	McLean County ⎫	Pit-run materials that do not meet Illinois grade 7 specifications	A-1-b(0)		NP	4
	Bureau County ⎬		A-1-b(0)		NP	<2
Ava B	Williamson County	Highly weathered loess	A-6(10)	39	15	28

parent material will be encountered in pavement construction only in moderately deep cut sections and in material that is excavated from borrow pits. Thus, a very large percentage of the surficial materials encountered in highway construction in Illinois consists of weathered material that is fine grained in texture.

Development of Sampling Program

The literature study (1) indicated that highly plastic, fine-grained soils can be most effectively and economically stabilized with lime. Cement can be used to stabilize these fine-grained materials, but, normally, the content required by current criteria (PCA) is quite high. Fine-grained soils throughout the state have been extensively sampled and tested in connection with lime stabilization research activities at the University of Illinois (4). Consequently, the sampling program did not include fine-grained materials.

With this background information and close coordination with Department of Agronomy, University of Illinois, personnel, a limited sampling program was developed in which representative Illinois parent materials (loess, Illinoian age drift, and Wisconsin age drift) and selected typical B-horizons were sampled. A general description and classification of the 14 coarse, medium, and moderately fine-textured soil samples are given in Table 1.

Laboratory Testing Program

A laboratory testing program was conducted to determine the strength and durability response of the 14 representative soils to treatment with various stabilization techniques. The techniques considered included conventional stabilization procedures (cement, bitumen, and lime-fly ash), blending techniques, and combination procedures (lime-cement and lime-bitumen). A discussion of the laboratory procedures and a summary of the data are presented elsewhere (2).

Based on the results of the laboratory program and the results of other research at the University of Illinois, both published (5) and unpublished, the tentative stabilization guidelines and criteria were reviewed, revised, and expanded to reflect the more specific characteristics of Illinois surficial soils and materials.

APPROACH USED TO APPLY STABILIZATION GUIDELINES AND CRITERIA TO ILLINOIS SOILS AND MATERIALS

In order to systematically apply the stabilization guidelines and criteria to the development of stabilization feasibility recommendations, it was necessary to identify and

classify soils of similar characteristics pertinent to soil stabilization and to collect necessary chemical and physical soil data.

A requirement of any classification system is that it be meaningful to the user. A number of classification procedures were examined to determine their relative merits. Of the currently available procedures, it was concluded that the pedological system provided the best method of soil classification for soil stabilization purposes. The potential of this system of classification in cement-soil stabilization has been demonstrated by Hicks (12), Leadabrand et al. (7), and Handy and Davidson (11).

Pedological Soil Classification System

The pedological classification system is based on the premise that a soil's structure, form, and properties are controlled by the extent of chemical and physical weathering to which a deposit has been subjected. A number of important environmental factors, often termed soil-forming factors, have been found to control the soil that is formed. The 5 soil-forming factors are type of parent material, relief, native vegetation, climate, and age or length of exposure.

Weathering of a parent material brought about by the various chemical and physical forces of the environment is most effective at the surface. The relative degree of weathering decreases with increasing depth from the surface in a manner such that various layers or horizons reflecting different stages of alteration are developed in the parent material. The horizons normally become thicker and more pronounced as soil development progresses. A vertical cross section that slices through the various horizons or layers is referred to as a soil profile. A typical soil profile is schematically shown in Figure 2 (6).

The concept of a soil profile and the variation of chemical and physical soil properties in the profile are very important to the development of stabilization recommendations. Soil properties such as texture, clay content, plasticity, pH, organic carbon content, and degree of oxidation vary in a given profile depending on the horizon. The A-horizon experiences maximum eluviation or leaching as a result of the relatively high degree of weathering. For this reason, the A-horizon normally contains relatively small amounts of soluble constituents and clay-sized particles. Normally, however, the organic matter content is rather high in the A-horizon because of the influence of local vegetation.

The B-horizon is the layer where illuviation or deposition of the material leached from the A-horizon occurs. Therefore, the B-horizon normally contains a much higher clay content and displays higher plasticity than the A-horizon; however, generally the organic matter content of the B-horizon is much lower. The C-horizon consists of relatively unaltered parent material and does not reflect the influence of the various chemical and physical weathering forces of the environment.

In some instances the C-horizon is not extremely thick and is underlain by a distinctly different parent material, well within the depth in which highway construction operations may occur. The nomenclature of this underlying layer of material varies from agency to agency.

As the degree of physical and chemical weathering changes as a result of various combinations of the soil-forming factors, profiles of differing characteristics result. The subtle differences that occur, however, are of great significance in the determination of stabilizer feasibility recommendations.

Identification and Classification of Soils in the Pedological System

A number of profile features are used by pedologists to describe a soil profile:

1. Number, thickness, and relative arrangement of horizons in the profile;
2. Organic matter content, usually reflected by the color of the horizon;
3. Drainage class, as influenced by slope, permeability, and position of water table;
4. Texture and structure of horizons;
5. Chemical and mineralogical composition;
6. Concretions and other special formations;
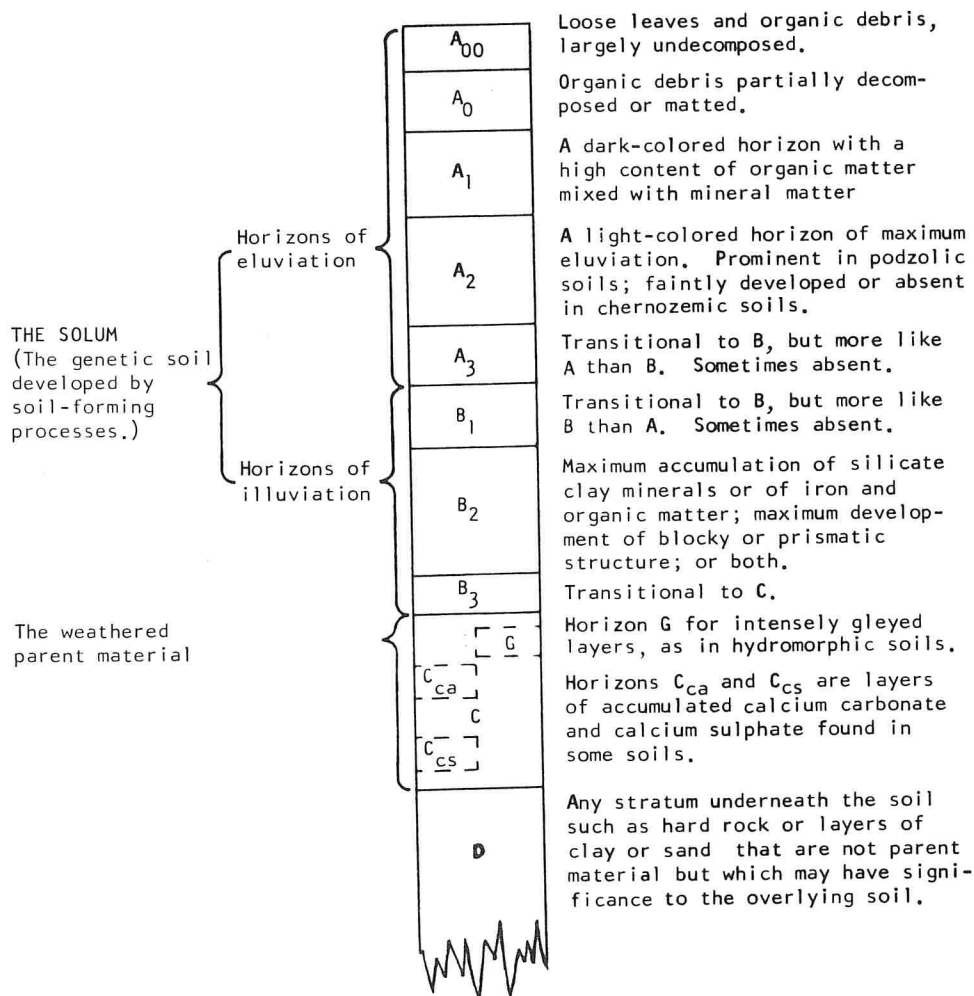
Figure 2. A typical pedologic soil profile showing the major horizons.

7. Vegetation; and
8. Geology of the parent material.

Soils that have profiles similar with regard to all of these characteristics are grouped together and form a soil type. All soil types with profiles having the same characteristics except for the texture of the surface horizon belong to the same soil series. Soils are further grouped into soil-association areas. A soil-association area is defined as a group of soils occurring together in a characteristic pattern (6). As mapped in Illinois on a statewide basis, a given soil association usually includes soil types and series developed from similar parent materials under similar vegetation conditions for approximately the same length of time (6).

Advantages of Using the Pedological Classification System

The pedologic system of soil classification offers a number of advantages in soil stabilization work. First, the basis of the pedological system is very pertinent because various chemical and physical soil properties are used as the classification criteria. Second, in most areas of the United States, extensive work has been done concerning

pedologic soil mapping and classification. As a result, excellent maps and pertinent physical and chemical data are readily available from county, state, and federal agencies. For example, extensive soil information was obtained for this research project from the Department of Agronomy, University of Illinois, and the Soil Conservation Service, U.S. Department of Agriculture.

Stabilization Technological Considerations

Effective and economical utilization of stabilization procedures requires that proper consideration be given to the type and nature of the stabilizing agents, properties of the soils and aggregates, stabilization objectives, and construction techniques.

For highway-related applications, stabilization objectives can be divided into 3 broad categories: construction expedient, subgrade modification, and strength and durability improvement.

Construction-expedient applications are concerned with improving vehicle-mobility problems often associated with fine-grained soils at high natural-water contents and degrees of saturation. In many cases, problems of this nature can be greatly improved or alleviated with the correct application of various stabilization techniques and procedures that effect an immediate improvement in (a) resistance to deformation, (b) load-carrying capacity, and (c) shear strength. In essence, a "working table" is created on which construction operations can be carried out in an efficient, effective, and economical manner.

Subgrade modification applications of stabilizing agents are concerned with improving engineering characteristics of the finer grained plastic subgrade soils. Stabilization of these materials will effect the following:

1. A decreased plasticity (some soils may actually become nonplastic with the amounts of stabilizer normally used);
2. A reduced swell;
3. An increased CBR, even though extensive cementing may not be obtained because of either a lack of "reactivity" (some soils do not display substantial strength gains when lime is added) or the use of stabilizer quantities less than required to effect substantial strength increases;
4. A decreased resilience;
5. An increased resistance to the detrimental effects of frost action, rainfall, or moisture content fluctuations; and
6. Improved workability characteristics and ease of handling.

Strength and durability improvement applications are concerned with upgrading (increased strength and durability) soils and aggregates to provide economical paving materials of subbase and base-course quality.

In the development of stabilization recommendations, appropriate considerations must be also given to field construction of stabilized layers and construction-related problems. Improper construction of stabilized materials can lead to unnecessary and costly failures of otherwise properly designed pavements.

Field construction techniques depend on the type of stabilizing agent being used, the type of soil being treated, the stabilization objectives, and, to a certain extent, the type of equipment available.

To obtain satisfactory field construction of stabilized materials, a number of factors, including pulverization, stabilizer distribution, mixing, blending, compaction, and curing, must be properly controlled to ensure the quality of the stabilized mixture.

ILLUSTRATIVE EXAMPLE

An example is an appropriate method of illustrating the type of stabilization recommendations that were developed and the format of presentation that was used. As an illustrative example, stabilization recommendations will be presented for the major soils of one of the 26 soil associations in which the soils of Illinois have been grouped by the Department of Agronomy, University of Illinois, and the Soil Conservation Service (8). Stabilization recommendations have been developed (9) for the major soils in all of the 26 soil associations of Illinois.

Characteristics of Soils in Soil Association B

Soil association area B occurs in east-central and north-central Illinois (Fig. 3) and occupies 2,631,000 acres or about 7.2 percent of the state. The soils in this association have developed under grass vegetation from either 3 to 5 ft of loess over loam to silty clay loam Wisconsin glacial till or 1 to 3 ft of loess over clay loam Wisconsin

Figure 3. General soil map of Illinois.

TABLE 2

PROPERTIES OF MAJOR

Number and Name of Major Soil Series	Area of State[a] (percent)	No. of Acres	Dominant Slope (percent)	Drainage	Soil Profile and Site Description[b]	Depth From Surface (in.)
171 Catlin (a)	1.15	377,100	3-7	W-MW	About 1 ft of silt loam on 2 ft of silty clay loam underlain by silt loam grading to calcareous loam glacial till. Well drained, nearly level to strongly sloping soils formed from about 3 to 5 ft of loess over calcareous loam glacial till.	0-12 12-42 42-60
56 Dana (b)	0.16	51,500	3-7	MW	About 1 ft of silt loam on 2½ to 3 ft of silty clay loam underlain by non-calcareous clay loam glacial till that becomes calcareous below 4 ft. Moderately well drained, nearly level soils formed from 2 to 4 ft of loess over glacial till.	0-14 14-50 50-60
152 Drummer (a and b)	6.06	1,991,100	0-1	P	About 1½ ft of silty clay loam on about 1½ ft of silty clay loam and underlain by silt loam, loam, or sandy loam glacial drift. Poorly drained, level soils on loess (40 to 60 in. thick) covered till plains and moraines or stream terraces and outwash plains. Seasonal water tables at or near the surface.	0-16 16-36 36-60
154 Flanagan (a)	3.31	1,086,700	1-3	Imp.	About 1 ft of silt loam on 2 to 3 ft of silty clay loam and underlain by loam to silt loam till. Somewhat poorly drained level to sloping soils on loess (40 to 50 in. thick) covered till plains and moraines. Seasonal water tables above 3 ft.	0-14 14-44 44-60
481 Raub (b)	0.14	45,800	1-3	Imp.	About 1 to 1½ ft of silt loam on 2 ft of silty clay loam on 1 to 2 ft of clay loam underlain by calcareous loam glacial till. Somewhat poorly drained, nearly level to gently sloping soils formed in about 1½ to 3 ft of loess on glacial till plains. Seasonal water tables are within 3 ft of the surface.	0-14 14-50 50-60
55 Sidell (b)	0.07	23,700	6-12	W	About 1 ft of silt loam on 1 to 1½ ft of silt clay loam on 1½ to 2 ft of clay loam underlain by loam glacial till. Well drained, gently sloping to moderately steep soils formed in 2 to 3 ft of loess on glacial till plains and moraines.	0-14 14-50 50-60

[a]From reference 10.   [c]Data supplied by Department of Agronomy, University of Illinois.
[b]From reference 3.   [d]Data from engineering publications, unpublished research in Department of Civil Engineerin

Note: Parent material—(a) Loess 3 to 5 ft thick on calcareous loam to silty clay loam till or (b) loess 1 to 3 ft thick on cl:

Soil Classification		Percent Passing Sieve[b]		Clay Fraction < 2μ[c] (percent)	Liquid Limit[d] (per-cent)	Plasticity Index[d] (percent)	Natural Soil pH[b]	Organic Carbon Content, Average Depths of Occurrence[c] (in.)		
USDA Tex-tural[b]	AASHO[b]	No. 4	No. 200					< 2 Per-cent	1-2 Per-cent	> 1 Per-cent
Silt loam	A-6 or A-7	100	95-100	15-25	30-50	10-25	5.6-6.5			
Silty clay loam	A-6 or A-7	100	95-100	30-40	35-58	15-30	5.6-6.5	0-16	16-22	22-60
Silt loam to loam	A-4 or A-6	95-100	55-75	15-25	30-55	11-20	7.4-8.4 (calc.)			
Silt loam	A-6 or A-7	100	95-100	15-25	30-55	10-25	5.6-6.5			
Silty clay loam to clay loam	A-6 or A-7	100	60-90	30-40	35-55	15-35	5.6-6.0	0-16	16-22	22-60
Loam to silt loam	A-4 or A-6	95-100	55-85	15-25	25-50	11-20	7.4-8.4 (calc.)			
Silty clay loam	A-7	95-100	85-100	30-40	35-65	15-33	6.6-7.3			
Silty clay loam	A-7 or A-6	95-100	85-100	30-40	30-55	12-34	6.6-7.3	0-16	16-24	24-60
Silt loam, loam, or sandy loam	A-2, A-4, or A-6	90-100	30-75	10-25	NP-50	NP-30	6.6-7.3			
Silt loam	A-6 or A-7	100	95-100	15-25	30-60	15-29	5.6-6.5			
Silty clay loam	A-6 or A-7	100	95-100	35-45	42-60	16-35	5.6-6.5	0-12	12-20	20-60
Loam, silt loam, or silty clay loam	A-6 or A-4	95-100	55-75	15-25	30-55	11-30	7.4-8.4 (calc.)			
Silt loam	A-6 or A-7	100	85-95	15-25	—	—	5.6-6.5			
Silty clay loam to clay loam	A-6 or A-7	100	65-95	35-45	—	—	5.6-6.5	0-12	12-20	20-60
Loam to silt loam	A-4 or A-6	100	60-70	15-25	35-50	11-20	7.4-8.4 (calc.)			
Silt loam	A-6 or A-7	100	95-100	15-25	30-55	10-25	5.6-6.5			
Silty clay loam to clay loam	A-6 or A-7	100	60-90	30-40	35-55	15-35	5.6-6.0	0-16	16-22	22-60
Loam to silt loam	A-4 or A-6	100	55-85	15-25	25-50	11-20	7.4-8.4 (calc.)			

loam till (noncalcareous > 3½).

glacial till. Various sources of information (3, 6, 10) and data supplied by the Department of Agronomy, University of Illinois, were used in preparing Table 2, which summarizes typical ranges in pertinent soil properties and characteristics of the major soil series within soil association B.

The basic differences recognized in mapping the soils in this association are parent material and internal drainage or slope. Sidell, Catlin, and Dana are well, well to moderately well, and moderately well drained soils respectively; Flanagan and Raub are imperfectly drained soils; and Drummer is a poorly drained soil.

As a result of being developed under grass vegetation, relatively thick A-horizons of relatively high organic matter content are characteristic of these soils. Because organic matter is known to be detrimental to many stabilization reactions inherent with "active" stabilizers, typical depths of occurrence for various amounts of organic carbon are given in Table 2 for the major soils of this association. As noted, the organic carbon content is typically greater than 2 percent to a depth of 12 to 16 in. and is less than 1 percent at depths greater than 20 to 24 in.

Available information indicates that the A-horizon materials are typically A-6 or A-7; the B-horizon materials, a more plastic A-6 or A-7; and the C-horizons, A-4 or A-6 depending on the specific parent material encountered.

The average plasticity index for the A-horizon materials normally ranges from 10 to 30; the materials of the B-horizon normally display a plasticity index ranging from 15 to 35; the plasticity index displayed by the C-horizon materials depends on the specific parent material but normally ranges from 10 to 30. The clay content (<0.002 mm) for the A-, B-, and C-horizons normally ranges from 15 to 25, 30 to 45, and 10 to 25 percent respectively (except that A-horizon of Drummer ranges from 30 to 40).

Stabilization Recommendations and Rating System

After collecting and summarizing the data given in Table 2, it was possible to apply the stabilization guidelines and criteria to the individual soils and determine stabilization recommendations for the major soil series of this soil association. These recommendations are given in Table 3. Recommendations are presented concerning the feasibility of using cement, lime, bitumen, lime-fly ash, and combination stabilizing agents for stabilization (to either expedite construction, modify the subgrade, or substantially

TABLE 3
STABILIZATION RECOMMENDATIONS
FOR MAJOR SOILS OF SOIL ASSOCIATION B

Number and Name of Major Soil Series	Horizon	Stabilization Objectives and Recommended Stabilizers[a]														
		Construction Expedient[b]					Subgrade Modification[b]					Strength and Durability Improvement[b]				
		C	L	B	F	COMB	C	L	B	F	COMB	C	L	B	F	COMB
171 Catlin	A	2	1	3	3	3	2	1	2	3	3	2	3	3	3	3
	B	2	1	3	3	3	2	1	3	3	3	1	2	3	3	—c
	C	2	1	3	3	3	1	1	3	3	3	1	1	2	2	—c
56 Dana	A	2	1	3	3	3	2	1	2	3	3	2	3	3	3	3
	B	2	1	3	3	3	2	1	3	3	3	1	2	3	3	—c
	C	2	1	3	3	3	1	1	3	3	3	1	1	2	2	—c
152 Drummer	A	2	1	3	3	3	2	1	3	3	3	2	3	3	3	3
	B	2	1	3	3	3	2	1	3	3	3	1	1	3	3	—c
	C	2	1	3	3	3	1	1	3	3	3	1	1	2	2	—c
154 Flanagan	A	2	1	3	3	3	2	1	2	3	3	2	3	3	3	3
	B	2	1	3	3	3	2	1	3	3	3	1	2	3	3	—c
	C	2	1	3	3	3	1	1	3	3	3	1	1	2	2	—c
481 Raub	A	2	1	3	3	3	2	1	2	3	3	2	3	3	3	3
	B	2	1	3	3	3	2	1	3	3	3	1	2	3	3	—c
	C	2	1	3	3	3	1	1	3	3	3	1	1	2	2	—c
55 Sidell	A	2	1	3	3	3	2	1	2	3	3	2	3	3	3	3
	B	2	1	3	3	3	2	1	3	3	3	1	2	3	3	—c
	C	2	1	3	3	3	1	1	3	3	3	1	1	2	2	—c

[a]Stabilizer suitability rating where 1 = suitable, 2 = questionable, and 3 = not suitable.
[b]C = cement, L = lime, B = bitumen, F = lime-fly ash, and COMB = combinations.
[c]Combination stabilization (lime-cement or lime-bitumen or both) may be a suitable method of beneficiation. However, this recommendation is based on a very limited amount of laboratory results and the respective literature surveys.

improve strength and durability) of the material in the A-, B-, and C-horizons of each major soil series.

Stabilization recommendations for a particular material carry either a 1, 2, or 3 rating. A 1-rating indicates that the stabilizing agent should quite suitably and adequately provide stabilization if proper construction practice is used. A 2-rating indicates a questionable rating because the stabilizing agent may provide adequate stabilization but, in certain cases, adequate stabilization cannot be obtained. A 3-rating indicates that, in most cases, the particular stabilizing agent will not provide the degree of stabilization desired.

Discussion of Stabilization Recommendations

Experience with oiled earth roads in Illinois has indicated that, in some cases, periodic treatment of organic A-horizon materials with cutback bituminous materials may provide a satisfactory pavement surface layer for low traffic volume roads. Stabilization of this nature was assumed to be in the realm of subgrade modification applications. A 2-rating was assigned as the bituminous recommendation for all A-horizon materials except those that are the very high clay content and high plasticity A-7 materials, such as the A-horizon of the Drummer soils. Treatment of these fine-grained materials, in most cases, will not provide a satisfactory surface layer, and, thus, a 3-rating was assigned.

Both cement and lime can be used in subgrade modification applications with the finer textured A-6 and A-7 materials of the B- and C-horizons. However, most data indicate that lime is more effective and efficient than cement. Consequently, lime has been assigned a 1-rating and cement a 2-rating. It is noted, however, for A-4 and low-plasticity A-6 C-horizon materials, that cement also has been given a 1-rating.

Cement can be used to obtain increased strength and durability with fine-grained A-6 and A-7 materials and, thus, a 1-rating has been assigned to these materials (B-horizon material of all major soils in this association). It should be realized, however, that the cement content required to obtain adequate durability for these fine-grained, plastic materials (PCA criteria) may be quite high, on the order of 9 to 16 percent (by soil dry weight).

Lime treatment of "reactive" fine-grained soils will effect substantial strength increases. A 1-rating has been assigned as the stabilization recommendation for lime where the soil properties indicate that reactive soils will be encountered. Many soils may display slight-to-moderate strength increases on treatment with lime, and, under certain service conditions, the lower strength lime-treated soils may be satisfactory. Thus, for those fine-grained materials that display limited reactivity, a 2-feasibility rating has been assigned with the supposition that, under certain service conditions, this can be changed to a 1-rating.

Stabilization recommendations have been presented concerning the applicability of combination procedures (lime-cement and lime-bitumen). A footnote reference is placed in Table 3 where it was felt that this procedure might be used. A 1-rating was not assigned because the rating is based on very limited data and a brief literature survey.

SUMMARY AND CONCLUSIONS

Stabilization recommendations for the major soils and materials occurring in Illinois have been developed (9). The general procedures and concepts used in the development of the stabilization recommendations and a typical example of the recommendations are presented in this paper.

In developing these stabilization recommendations, the pedological soil classification system was used to identify and classify soils of similar characteristics relative to soil stabilization. The soil association area concept was used to combine pedologic soil series into groups with similar characteristics and to facilitate presentation of pertinent data and feasibility recommendations.

Extensive soil data necessary to properly apply the stabilization guidelines and criteria were obtained from the Department of Agronomy, University of Illinois, the Soil

Conservation Service, the University of Illinois Engineering Experiment Station publications, and various other sources of published and unpublished data.

Although stabilization recommendations were presented only for major soils within each soil association area, by utilizing the guidelines and criteria that were developed for the soils of Illinois and by appropriate consideration of stabilization technology, stabilization feasibility recommendations can be readily determined for other soils.

The information that can be obtained from a report containing stabilization recommendations of this type is meant to facilitate the selection of a stabilizing agent or agents and to promote maximum use of local materials in various highway construction applications. However, additional factors must be considered in order to obtain quality stabilized materials meeting job requirements. Appropriate mixture design procedures must be used to satisfy job requirements for particular applications. Economic considerations, in many cases, may be very important and will often be used in the final selection of a stabilizing agent. Finally, proper construction techniques and control must be used to ensure that a quality product is obtained in the field.

It has been demonstrated that, by using the extensive soil information that is available from various sources, stabilization recommendations for large areas, such as the state of Illinois, can be developed with a minimum amount of sampling and testing. The concepts demonstrated in this paper can be used to develop stabilization recommendations for areas of other sizes such as residential areas, cities, counties, and regions.

ACKNOWLEDGMENT

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Experimental Stabilization of Pierre Shale

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The experimental study of stabilization of the Pierre shale was initiated with a threefold purpose in mind: (a) to determine if it is possible to achieve the degree of stability necessary to prevent warping of road surfacing materials by the addition of chemicals or other additives to the highly expansive in-place soils over which these surfacing materials are placed; (b) to determine if the use of additives in the in-place soils is more effective in reducing warping than replacing with nonexpansive materials; and (c) to determine, to a certain degree, the economy of using stabilizing agents in the in-place soil as compared to hauling in nonexpansive material. The experimental road includes the following types of stabilization in the upper 6 or 9 in. of soil subgrade: lime stabilization, lime-asphalt stabilization, phosphoric acid plus ferric sulphate, and PDC formula (4 parts lime, 2 parts cement, and 1 part soy flour). Control sections are composed of standard design methods, including select soil, or nonexpansive soil, varying in thickness from 6 to 18 in. in conjunction with a 3- to 6-in. thickness of standard type B subbase, 5 in. of standard type II base course, and a 2-in. class F mat. One special control section has 30 in. of select soil. Two other control sections have the earth subgrade undercut to a depth of 36 in. The undercut soil was replaced and recompacted prior to placement of surfacing materials.

At the end of the 4-year study period, all of the stabilizing agents have altered the physical characteristics of the Pierre shale to some degree. Lime appears to have the more permanent effect. The serviceability index ratings indicate that all of the stabilized sections except phosphoric acid are better than in the standard design sections. All of the treated soils have a higher CBR value after a 4-year period than the raw soil. The phosphoric acid section is only slightly higher than the raw soil. Lime-treated soils, and additives in combination with lime, apparently recover some bearing strength after suffering a loss of strength due to freezing. Serviceability index ratings indicate that treated soils tend to be more effective in reducing surface warping than the use of nonexpansive soils over the Pierre shale. It appears that the use of stabilizing agents capable of maintaining a permanent change in the physical characteristics of the soil would be more economical than the use of nontreated soils by reducing the long-range maintenance costs. Stabilizing agents capable of maintaining higher stability and bearing capacity in the treated layers throughout the year will allow the design of thinner surfacing components.

•WESTERN SOUTH DAKOTA and a portion of the state east of the Missouri River lie within the Great Plains Physiographic Province. The portion of the state within the James Basin and eastward lies in the Central Lowlands Province. The western portion of the state is divided into 3 general landforms, composed of the following:

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1. The Black Hills, a large domal uplift area that extends northwestward across the southwestern part of the state with elevations up to 7,200 ft;

2. The High Plains area in the south-central part of the state, extending along the South Dakota-Nebraska boundary at an elevation of 3,000 ft; and

3. The Missouri Plateau, a region of relatively low undulating grasslands that surrounds the Black Hills and the High Plains. This area contains the Badlands of South Dakota and the Pierre shale deposits.

The eastern portion of the state is also divided into 3 general landforms as follows:

1. Missouri Hills, in the central portion of the state;

2. James Basin, principally a low-lying, prehistoric lake bed in the north; and

3. The Prairie Hills, an area extending from the east central Minnesota border to the James Basin.

These physiographic divisions are shown in Figure 1.

Much of the soil in the Missouri Plateau in the western portion of the state is composed of Pierre shale, which exhibits highly expansive properties. This expansion causes considerable problems in building and maintaining smooth road surfaces. Our test road was built with the primary purpose of determining how effective any of several known chemical stabilizations would be in reducing surfacing roughness that develops soon after hard surface roads are placed on the expansive shale.

Because of the extremely high liquid limits of the Pierre shale, we were skeptical that any type of stabilizing agents could be adequately mixed in the field to provide any degree of uniformity. Although we were quite successful in achieving a good mixture of the high liquid limit shale-clay with various additives in the laboratory, we were not certain that this degree of uniformity could be duplicated in the field. However, warping of surfacing materials is so acute, in the expansive soil areas, that we felt that we had to try some remedial measures.

Experimental laboratory testing was completed in 1960, and we made plans to go ahead with a test road project. Shortage of state funds delayed the construction of the project, and it was not until 1963 that we were able to let a contract. All phases of the construction work were completed in September 1964.

It is the intent of this report to make rational, valid conclusions based on results of test data representing the various types of stabilization as they relate to the field conditions in South Dakota.
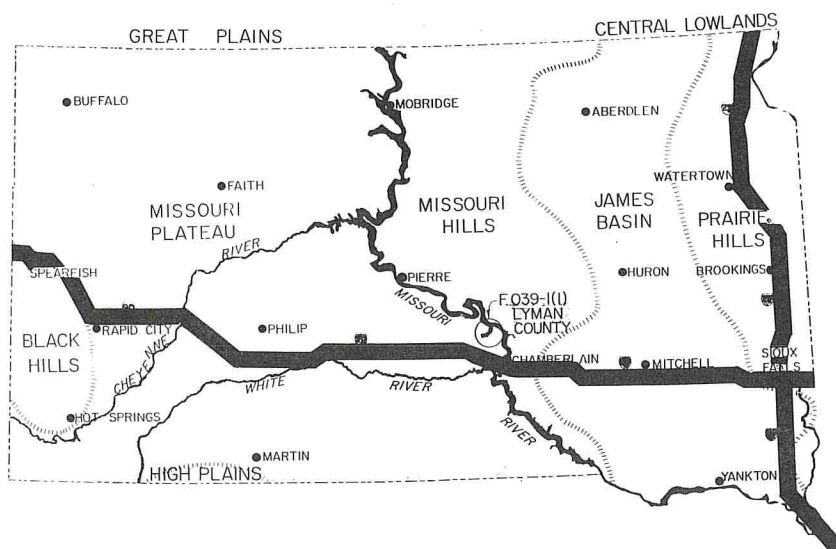
Figure 1. Physiographic divisions of South Dakota.

OBJECTIVES

The primary objectives of the test road are as follows:

1. To determine if it is possible to achieve the degree of stability necessary to prevent warping of road surfacing materials by the addition of chemicals or other additives to the highly expansive in-place soils;

2. To determine if the use of additives in the in-place soils is more effective than replacing with nonexpansive materials; and

3. To determine, to a certain degree, the economy of using stabilizing agents for the in-place soils, as compared to hauling in nonexpansive material.

In order to determine if it was possible to obtain results that would achieve these objectives the following stabilizing agents were introduced into the subgrade: lime stabilization, lime-asphalt stabilization, phosphoric acid plus ferric sulphate, and PDC formula (4 parts lime, 2 parts cement, and 1 part soy flour).

Control sections used for comparison purposes are composed of a 2-in. class F mat and a 5-in. base course, in conjunction with 3 to 6 in. of standard subbase. Several of these sections also had a layer of select soil varying in thickness from 6 to 18 in. placed below the subbase.

It is difficult to make an economic study of the cost of the individual types of stabilization on a project of this nature because relatively small quantities of some agents were used. However, maintenance cost records have been kept, and an effort has been made to compare the initial costs of the various types of stabilization against the indicated long-range maintenance costs. The initial cost of the standard design was $67,500 per mile, lime was $85,200 per mile, lime plus RC-1 was $93,300 per mile, PDC was $95,700 per mile, and phosphoric acid plus ferric sulphate was $120,600 per mile. These were the actual bid prices.

Laboratory Procedure

Laboratory tests were run on samples of the highly expansive Pierre shale taken from the F 039-1(1), Lyman County Project on SD-47W, which links the east river country and the west river country via the Big Bend Dam. The Pierre shale becomes very hard and slate-like when dried, and it is given a pretreatment breakdown with a wooden mallet
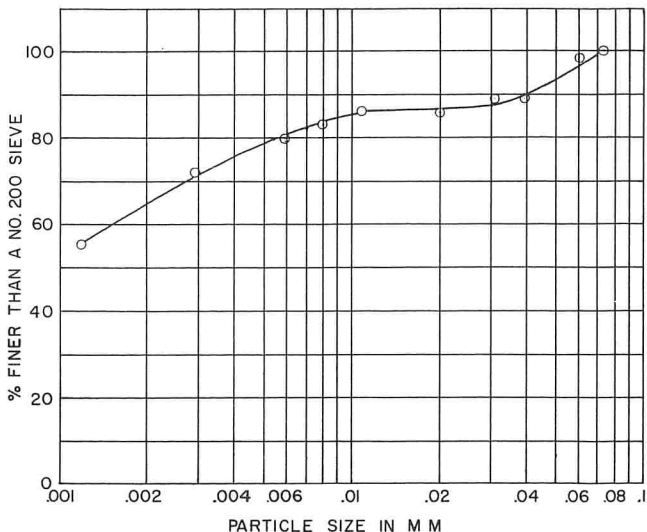
Figure 2. Particle size distribution of South Dakota shale.

prior to running it through a jaw crusher. The soil is further pulverized in revolving barrels in which rubber covered steel rollers have been placed. These rubber covered steel rollers are very effective in reducing the hard shale to a very fine grain size. This type of pulverization facilitated the mixing of lime, oil, phosphoric acid, ferric sulphate, and PDC and allowed a reduced curing period. The particle size distribution of the Pierre shale is shown in Figure 2.

A mechanical analysis of the soil was determined using the standard AASHO T-88-57 method. Hydrometer tests and the standard Atterberg AASHO T-89-60 and T-90-61 liquid limit and plastic limit tests were made. A sufficient amount of soil was prepared in all of the original samples to run standard AASHO T-99 densities, standard CBR tests, unconfined compression tests, and volume-change tests for both the raw samples and the treated soil samples.

To be sure that the amount of material was the same for each soil type specimen molded, with the various percentages of additives, a predetermined maximum density was used. Although nearly all of the tests made prior to construction showed that treated soils had a decided increase in unconfined compressive strengths, we were not able to extract these small undisturbed cores during post-construction testing to compare with the original laboratory results. Consequently, we had no data for this item in 1965. This test was replaced with a modified laboratory CBR by extracting 6-in. diameter cores and testing them under the same conditions as the laboratory molded CBR's.

The effects of compactive effort are shown in Figure 3, using the modified AASHO method and the standard AASHO method. The Pierre shale is quite sensitive to compactive efforts. It can be seen that there is a 17.5 lb/cu ft weight differential and a corresponding moisture differential of 10 percentage points when modified AASHO T-189 is used as compared to standard AASHO T-99. Figures 4 and 5 show that expansive pressures of raw soil are affected by both compactive effort and moisture content.
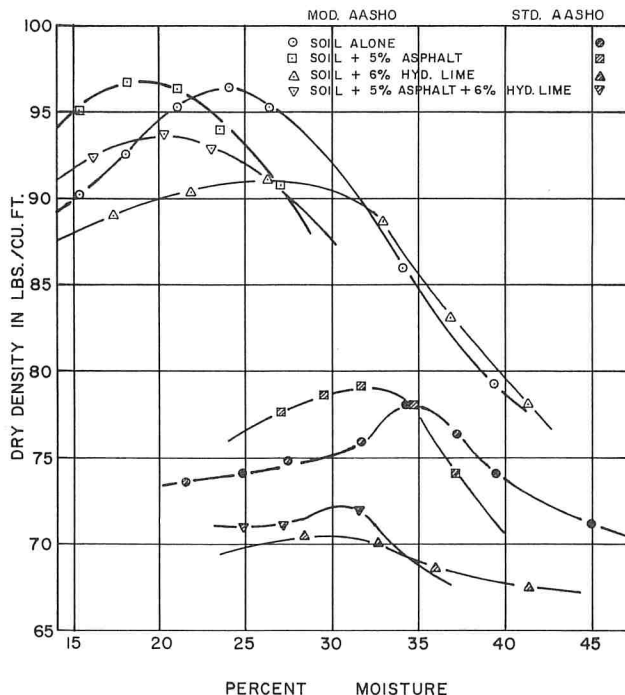
Figure 3. Effect of compactive effort on density of South Dakota shale.
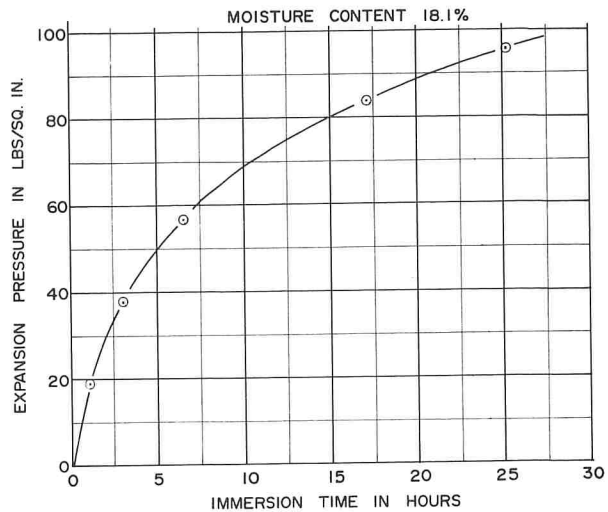
Figure 4. Expansion pressures of South Dakota soil—Modified
AASHO compaction T-180.
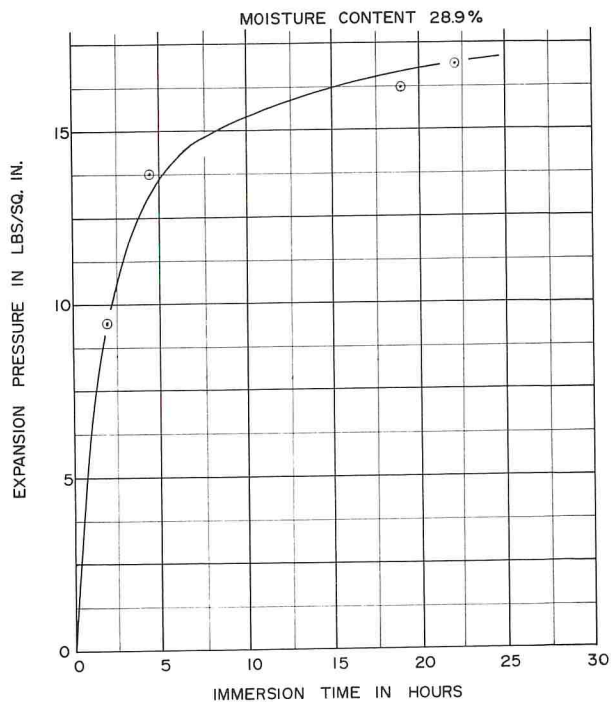
Figure 5. Expansion pressure of South Dakota soil—Standard
AASHO compaction T-99.

In addition to these tests described, a series of freeze-thaw tests were conducted on samples containing the various types of additives to determine what effect freezing would have on the stabilized soil. One set of specimens for each type of stabilization was frozen and allowed to thaw out in the air at room temperature. The other set was frozen and thawed out under water at room temperature. We found that all of the stabilization types suffer quite a bit of damage after 12 freeze cycles. The difference in the degree of deterioration of specimens within each stabilization type indicated that variation in laboratory procedure and technique have some effect on how well the specimens withstand the freezing cycles. Figure 6 shows that some specimens deteriorated completely and other stood up quite well when thawed at room temperature. Figure 7 shows the results of the soak thaw.

Our previous experience indicated that the expansive soil produced warping effects on either concrete or asphalt surface roads. However, we included approximately 1 mile of concrete surfacing as well as a short section of deep-strength asphalt and 1 short section of cement-treated base with an asphalt overlay. A cement-treated base was also placed under portions of the concrete surfacing. These surfacing types were introduced for observation purposes and to determine the effects of warping on the riding quality of the surfacing.

Field Procedure

Preconstruction—Prior to the beginning of stabilizing the subgrade, the service gravel that had been placed to carry local traffic had to be removed. After this operation there were still a few gravel particles remaining in the upper 6 in. of roadbed. These particles were screened out and ignored during the molding operations of samples taken from the roadbed.

Construction—The average liquid limit of the soil through the first 6 miles of the test section is approximately 91 percent. The average optimum moisture is 30 percent. The last 2.5 miles of project has an average liquid limit of 61 percent and an optimum moisture of 24 percent.
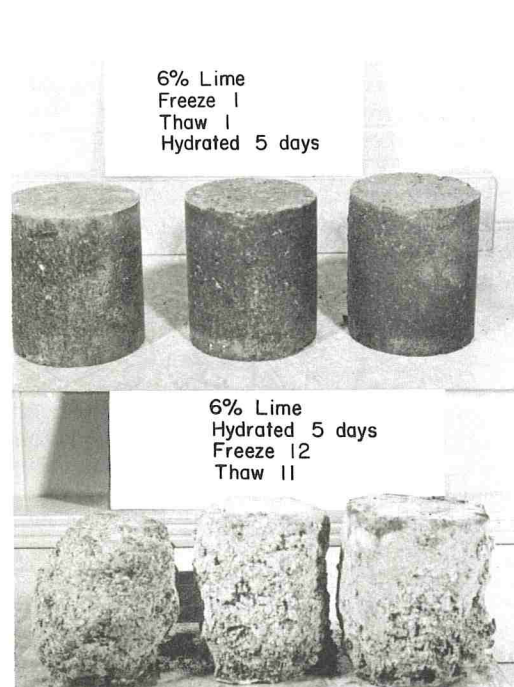
Figure 6. Samples after air thaw.
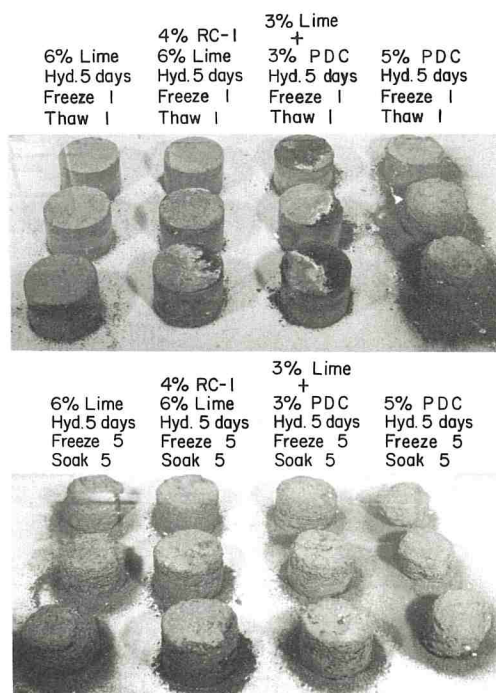
Figure 7. Samples after soak thaw.
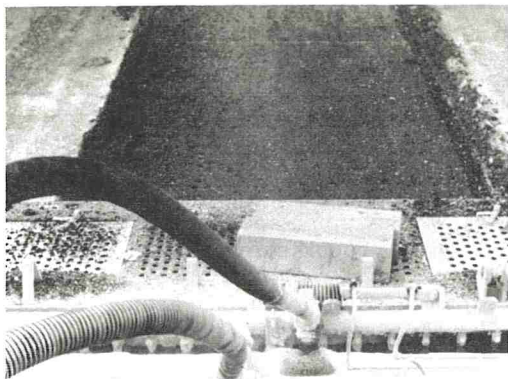
Figure 8. Single-pass pulverizing machine.

Figure 9. Project identification markers.

Specifications required that the soil should be thoroughly pulverized prior to addition of the additives with any type of single-pass machine that the contractor believed would produce the desired results. It was required that not less than 95 percent of the material would pass the $1\frac{1}{2}$-in. sieve and not less than 50 percent would pass the $\frac{3}{4}$-in. sieve. The contractor used a single-pass machine; and, although he had to move at a slow speed, pulverization specifications were met in a single pass (Fig. 8). This greatly facilitated the progress of the stabilization work.

The roadbed was treated with the stabilization materials during the spring of 1964, and the surfacing materials placement was completed during September 1964. Benchmarks are set along the right-of-way at locations convenient to the various stabilized sections in order to establish precise level data in connection with the rideability and roughness study.

Upon completion of stabilization work, identification markers were placed at the ends of the project and at the end of each type of stabilization and control sections. These markers at each test section are a graphic, coded picture of the thickness and type of each surfacing and stabilizing component used in each test section (Fig. 9).

POST-CONSTRUCTION TESTING

Laboratory Tests and Procedures

The standard AASHO T-89-60 test was used to determine the liquid limit of the treated and raw soils. These results are tabulated on a yearly basis and are compared to the original liquid limits determined during preconstruction and during construction. Figures 10 through 13 show that the average test results of all of the treated soils show some tendency to rebound or move upward to a higher value when compared to the original construction tests.

The plasticity index (PI) tests were run in accordance with standard AASHO T-90-61. There is a tendency for the PI's to rise slightly above the PI's noted during construction. However, the lime has held very close to the original treated PI's, and is slightly better than the lime plus RC-1, which is second best. The PDC pretreated with lime is third. The phosphoric acid plus ferric sulphate returned almost to the original PI. The average test results of each of the sections are shown in Figures 14 through 17.

Volume-change tests run on treated soils during construction show that all of the treated soils have considerably less volume change than the untreated raw soils. CBR swell tests for raw soils and treated soils, as compared with the modified CBR swell tests run on samples taken from the field during 1966, 1967, and 1968, show that the swell properties of all of the treated soils taken from the field compared favorably with
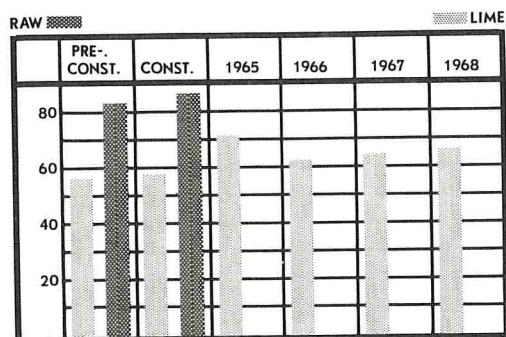
Figure 10. Liquid limit, lime stabilization.
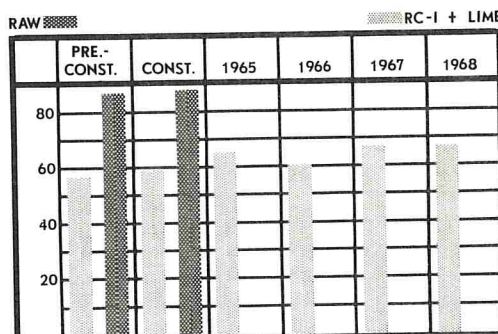
Figure 11. Liquid limit, lime-asphalt stabilization.
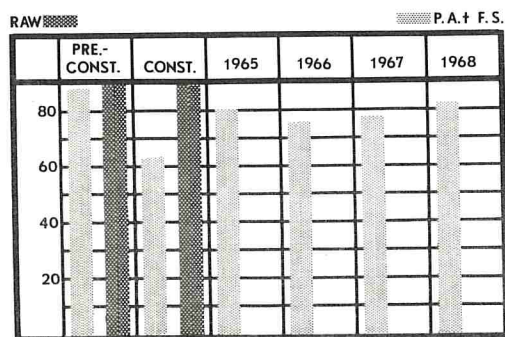
Figure 12. Liquid limit, phosphoric acid plus ferric sulphate stabilization.
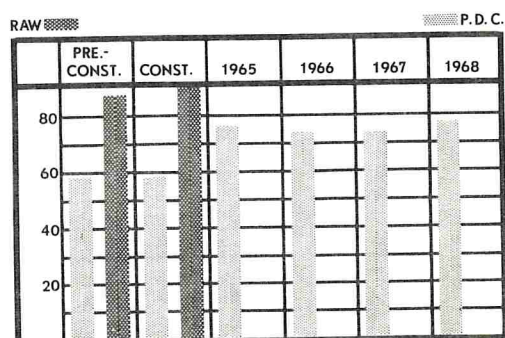
Figure 13. Liquid limit, PDC formula stabilization.
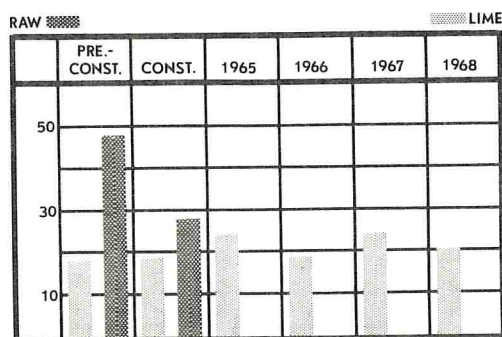
Figure 14. Plasticity index, lime stabilization.
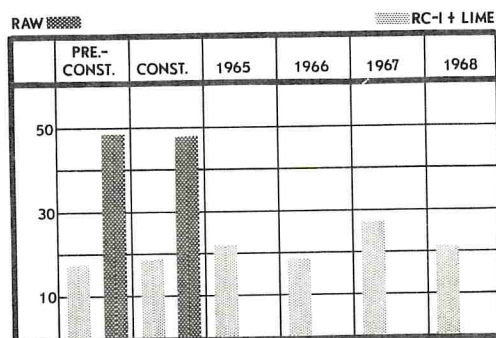
Figure 15. Plasticity index, lime-asphalt stabilization.

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the preliminary laboratory tests that were run prior to construction and during construction. Also, all of the treated soils have swell values considerably below those of the raw soils. The average results of each of the various test sections are shown in Figures 18 through 21.

In 1967 and 1968 we ran some pH tests to determine how much calcium oxide was still available for reaction with the soil. The pH of the raw soils varies from about 5.9
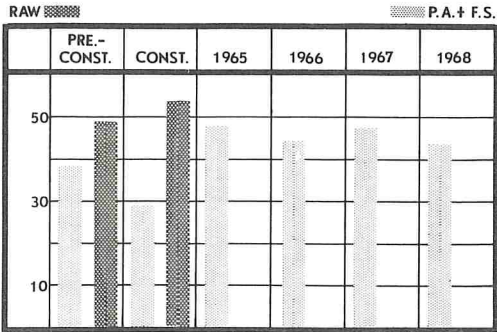
Figure 16. Plasticity index, phosphoric acid plus ferric sulphate stabilization.
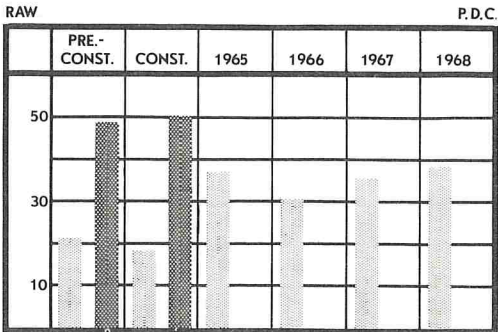
Figure 17. Plasticity index, PDC formula stabilization.
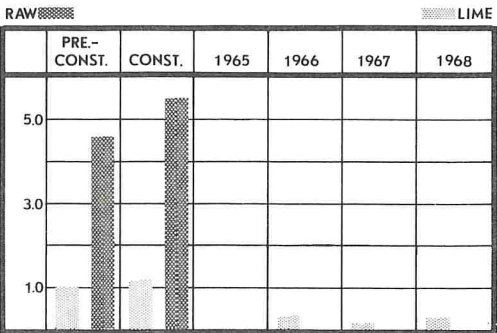
Figure 18. Percentage CBR swell, lime stabilization.
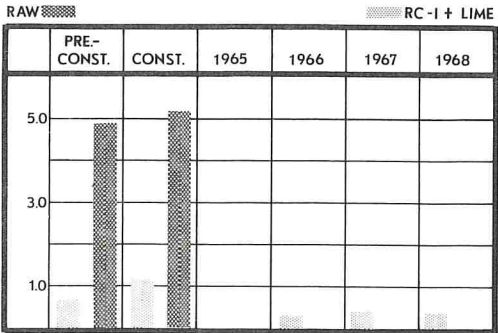
Figure 19. Percentage CBR swell, lime-asphalt stabilization.
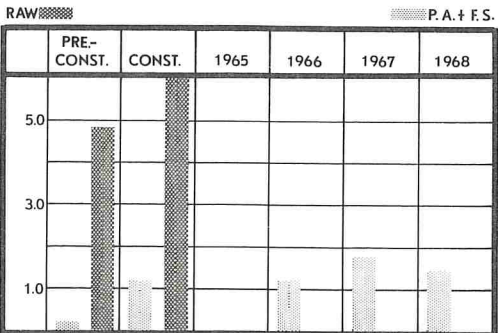
Figure 20. Percentage CBR swell, phosphoric acid plus ferric sulphate stabilization.
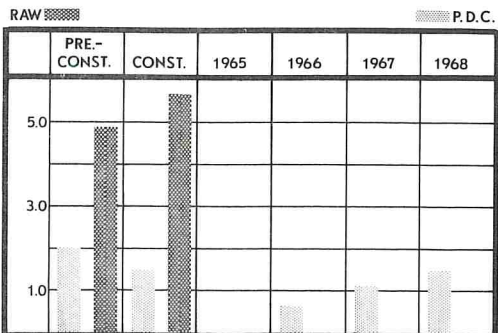
Figure 21. Percentage CBR swell, PDC formula stabilization.

to 8.2. We found that almost all of the treated sections, with the exception of the acid-treated soil, have a pH of 9 to 11. One PDC section, one lime section, and one lime plus RC-1 section show very little if any calcium oxide remains.

Field Tests and Procedures

We had planned to run our post-construction tests at the rate of 4 times per year, during each season of the year. Because of limited equipment and personnel, it was not possible to adhere to this schedule of testing. We were forced to sample and test the in-place materials on the basis of once a year through the period of approximately April 15 to October 15.

Stationing of the test sites in each test section was selected, and sampling operations were begun in April 1965. The Physical Research Section of the Department of Highways conducted standard plate tests and field CBR tests during the spring and summer months. A series of plates varying from 9 to 18 in. in diameter were used, and loads were applied to the mat as well as to each layer of surfacing, including the stabilized subgrade soil. In order to perform these tests, it was necessary to cut a series of 5 holes at each test site. The average test results of the 18-in. plate loads are shown in Figures 22 through 25.

In order to obtain samples of the treated subgrade for laboratory testing and field tests, it was necessary to bore 3 holes into the surfacing at each test site. A thin-walled, Shelby tube was forced into the exposed subgrade of one of the holes to a depth of 2 ft to obtain laboratory samples. A second hole was used to obtain an in-place specimen on which to conduct a modified CBR test and swell test. The third hole was
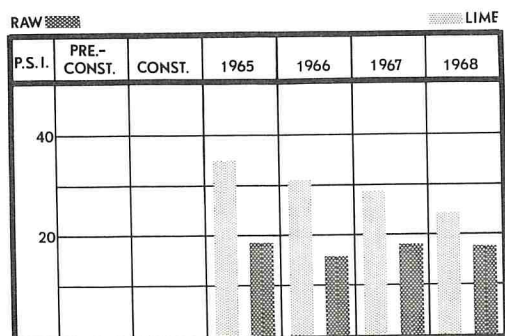
Figure 22. Plate load, lime stabilization.
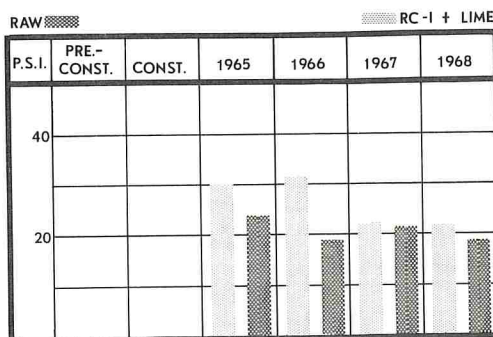
Figure 23. Plate load, lime-asphalt stabilization.
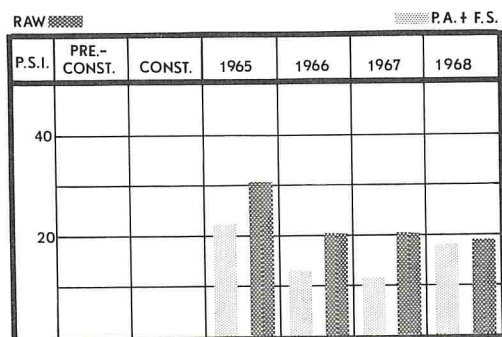
Figure 24. Plate load, phosphoric acid plus ferric sulphate stabilization.
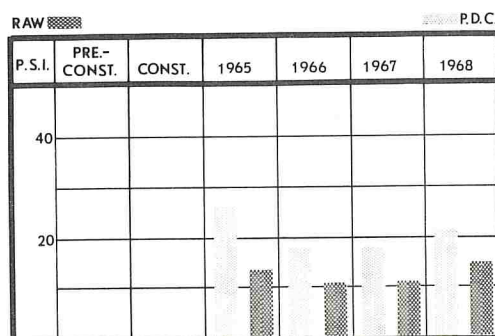
Figure 25. Plate load, PDC formula stabilization.

Figure 27. Fault lines and bentonite layers below roadbed.

Figure 26. Cracks that developed in areas of bentonite soil.

used to conduct a modified 6-in. plate load test. During the fourth year a fourth hole was dug to perform a modified field CBR test.

Large cracks developed where concentrations of bentonite soil are present beneath the roadbed. Investigations of these areas showed fault lines and nearly pure bentonite layers as far down as 15 ft below the roadbed, as well as free water accumulation (Figs. 26 and 27). A study of the crack pattern shows the average number of cracks per mile in the lime sections varied from 83 in 1966 to 145 in 1968. Those in the phosphoric-acid sections varied from 46 in 1966
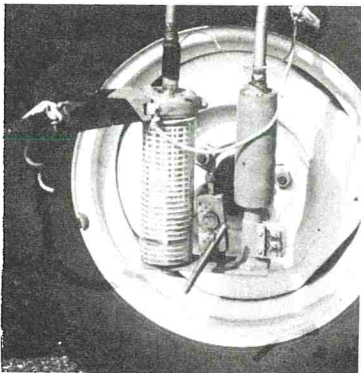
96A
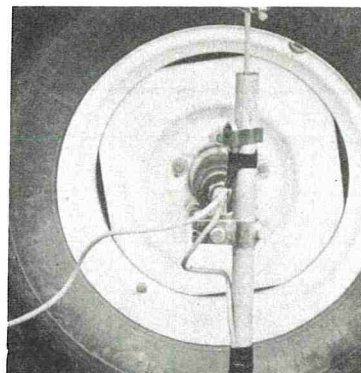
96B

Figure 28. Old roughometer (left) and new LUDT coil roughometer (right).

to 143 in 1968. The lime-plus-RC-1 crack pattern varied from 85 in 1966 to 147 in 1968. The cracks in the PDC sections varied from 101 in 1966 to 211 in 1968. The raw sections had a crack pattern range from 126 in 1966 to 211 in 1968. The cracks in the cement-treated base sections varied from 122 in 1966 to 550 in 1968. With the exception of the PDC and cement-treated bases, the treated soils definitely have some influence on the crack pattern of the surface.

CBR tests indicate that the lime-treated soils in nearly all test sections have considerably higher bearing values than the raw soil for both the soak CBR and the modified field CBR. The lime plus RC-1 rates second best, the PDC material is third, and the phosphoric acid is only slightly better than the raw soil for both types of tests.

In order to determine how the various degrees of warping were affecting the rideability in each test section, a high-speed roughometer was developed that travels the legal speed limit of 70 mph. This roughometer traces the wheel movement on a paper tape and also is recorded on a magnetic tape for introduction into a digitizing unit. The degree of displacement was recorded by setting permanent bench marks adjacent to the roadway and driving bolts in the road surface. Permanent marks were cut into the concrete. The bolts and marks were set at 25-ft intervals on centerline and in each outside driving lane. Precise level data were recorded 4 times a year at the seasonal changes through the 4-year period.

Total displacement, or average displacement, is not always indicative of the rideability or serviceability. However, when the displacement levels are extremely differentiated, the roughometer graph and the displacement graph correlate quite well. Figure 28 shows the old roughometer on the left and the new LUDT coil roughometer on the right. Figure 29 shows formulas for determining SI, and Figure 30 shows an example of the displacement as determined by the precise level data.

EXAMPLE OF WAVE FORM GENERATED BY THE ROUGHOMETER

S.I. IS COMPUTED AT EACH PEAK AND VALLEY AND THE AVERAGE S.I. DETERMINED AT THE END OF EACH SECTION.
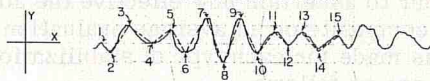
ROUGHNESS COMPUTED

$$\left[\frac{(Y_2-Y_2)}{.01(X_2-X_1)} - \frac{(Y_3-Y_2)^2}{.01(X_3-X_2)}\right] \frac{1000}{(X_3-X_1)} = R$$

SERVICEABILITY INDEX

IS COMPUTED TAKING INTO ACCOUNT CRACKING, PATCHING AND RUTTING.

ROUGHNESS INDEX

$$R.I. = \frac{125\,R}{R+25}$$

RIGID PAVEMENTS

$$S.I. = R.I. - 0.09\sqrt{C+P}$$

FLEXIBLE PAVEMENTS

$$S.I. = R.I. - 0.01\sqrt{C+P} - 1.38(RD)^2$$

In which:

R.I. = Roughness Index.
S.I. = Serviceability Index
C = Major cracking in ft. per 1,000 sq. ft. of area.
P = Bituminous patching in sq. ft. per 1,000 sq. ft. of area.
RD = Average rut depth of both wheelpaths in inches measured at the center of a 4 ft. span in the most deeply rutted part of the wheelpath.

Figure 29. Formulas for determining SI.
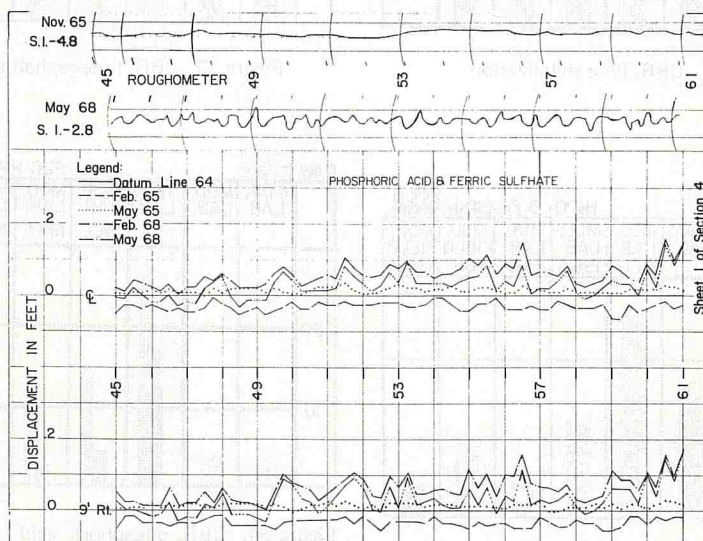
Figure 30. Example of displacement.

OBSERVATIONS

In order to ascertain how effective the additives are in achieving the objectives of the study, a composite or an average evaluation of the various tests that relate to these items was made for each type of stabilization. The evaluations for each of the test sections are as follows:

Lime-Treated Subgrade—A review of the test data shows the PI for the average of all lime sections has been reduced from 44.0 to 16.0. The average CBR shows a loss in value during the second and third year and then regains some of the initial strength. The modified field CBR value in 1968 is higher than the average laboratory CBR value in 1964 (Fig. 31).

Lime- and RC-1-Treated Subgrade—The PI of the treated soil during the 4-year study averages approximately 22.5 as compared with an average PI of 50.7 for untreated soil. The average CBR of the lime plus RC-1 soils shows the same trend as the lime-treated soil, namely, a loss of strength during the second and third year with a regain of strength in 1968. The modified field CBR also shows a higher value in 1968, being nearly equal to the original laboratory CBR (Fig. 32).

PDC-Treated Soil—The average plasticity index of the treated soil during the 4-year study period is maintained at approximately 32.0 as compared with a plastic index of approximately 50.4 for the untreated soil. The average CBR values show a loss during the second and third year of freeze and thaw and show only a small recovery. However, the modified field CBR of the treated soil is considerably higher in 1968 than that of the untreated soil (Fig. 33).
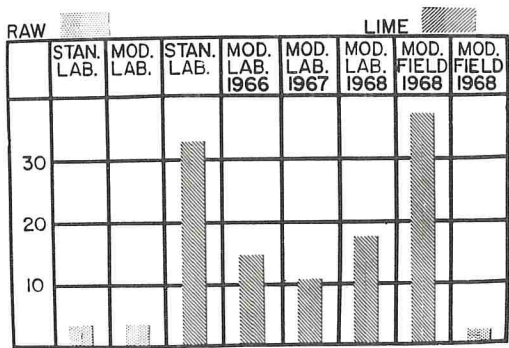
Figure 31. CBR, lime stabilization.
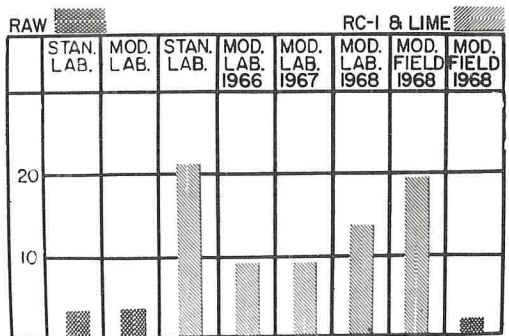
Figure 32. CBR, lime-asphalt stabilization.
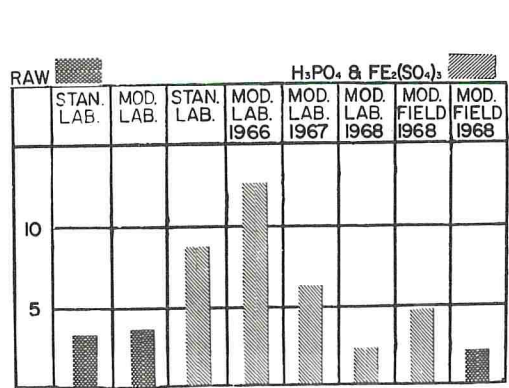
Figure 33. CBR, PDC formula stabilization.
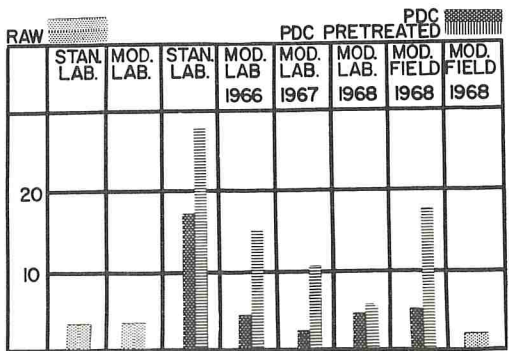
Figure 34. CBR, phosphoric acid plus ferric sulphate stabilization.

Phosphoric Acid plus Ferric Sulphate—
The average plasticity index of the treated
soil during the 4-year study is approxi-
mately 44.0 as compared with an approxi-
mate average plastic index of untreated
soil of 53.0. The modified laboratory CBR
values have dropped to almost the value of
the untreated soil. However, the modified
field CBR shows there is a slight retention
of strength when compared with the un-
treated soil (Fig. 34).

The overall average comparison of ser-
viceability index for cuts and fills for all
of the treated soils is shown in Figure 35.

CONCLUSIONS

More than 5,200 laboratory and field
tests were run on the treated and untreated
soils during the course of this study. More
than 95,000 elevation readings of the road
surface were taken.

It is important to understand that, al-
though all of the soils within the test road
area have an AASHO classification of
A-7-6(20), the physical characteristics, in
regard to liquid limit, vary from a low of
65 to a high of 118. The plastic index,
shrinkage, and volume change vary in the
same degree of magnitude. These varia-

Figure 35. Serviceability index, all treated materials.

ble conditions can and do take place within a distance of 20 ft. These variations are
the cause of variable test values obtained in some of the test sections by virtue of the
fact that the percentage of additives was not adequate to completely stabilize the ex-
tremely high liquid limit soil.

Our interpretation of the data is therefore based on the average conditions in regard
to the ability of the various stabilizing agents to accomplish the objectives as set forth
in the foregoing portion of this report. These conclusions are as follows:

1. All of the stabilizing agents have altered the physical characteristics of the high
liquid limit soil to some degree. The field condition tests indicate that lime has a more
permanent effect in altering these characteristics than the other combination additives
used in this test road.

2. The serviceability index ratings, as determined by the applied formula used in
this report, indicate that all of the stabilized sections, except the acid, are better than
the standard design sections. The average SI ratings show that lime-treated sections
have the best ratings followed very closely by the PDC and lime plus RC-1 stabilization.
The phosphoric acid plus ferric sulphate rating is slightly below that of the untreated
soil.

3. The sections having lime or lime combinations show a loss of CBR strength when
compared with the initial laboratory CBR during the second and third year with a re-
covery of some bearing strength during the fourth year. The sections with lime only
have the best CBR value after a 4-year, in-service period, with the lime plus RC-1 be-
ing second and the PDC formula being third best. CBR values of the phosphoric acid
are about the same as the untreated soils.

4. The recovery of bearing in the lime-treated soils appears to confirm the findings
of Dr. Marshall Thompson, University of Illinois, in regard to the autogenous healing
theory of lime-treated soils. Dr. Thompson's laboratory tests show that lime-treated
soils tend to recover bearing strength after suffering a loss of bearing due to frost ac-
tion (1).
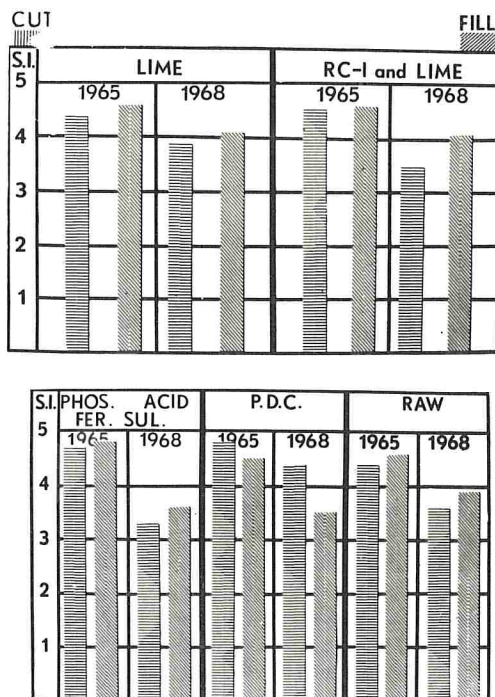
5. A review of the serviceability index indicates that the use of additives capable of maintaining a permanent change in the physical characteristics of the treated soil tend to be more effective in reducing surface warping than the use of nonexpansive regular material over the expansive soils.

6. As noted earlier in the report, the cost per mile was computed from the actual bid price for various stabilizing material. The prices are no doubt higher than they would normally be if large quantities of the materials were being purchased.

The fact that there are less cracks per mile in some of the treated sections, as compared with the untreated soil, indicates that the future long-range maintenance will be less. If the serviceability index differential between the lime and lime combination soils and the untreated soil continues at its present ratio, it appears that the future maintenance work in these sections will be delayed for a greater period of time than in the untreated sections.

7. In view of the fact that the stabilized subgrade maintains a higher stability and bearing capacity throughout the year, we are able to design thinner surfacing components, which allows us to conserve our dwindling gravel supply.

We believe that one of the big benefits derived from this study is the development of the high-speed roughometer, which is now being used as an aid in determining maintenance needs. We are in the process of developing and maintaining a yearly serviceability index rating on all Interstate, primary, and secondary routes. These data will be used to determine more precisely what the actual conditions of the road surfaces are and furnish the Maintenance Division with more realistic data on which to determine the type and amount of maintenance necessary for any given project. A report outlining the development, operation, and application of this roughometer will be written and published.

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Field Studies on the Pulverization of Black Cotton Soil for the Construction of Stabilized Soil Road Bases

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Stabilization of black cotton soil with lime has been found effective in improving the engineering properties of the soil. Consequently, this has led to the increasing use of lime-stabilized black cotton soil in subbases or bases of road pavement. A properly pulverized soil is, however, a prerequisite for successful stabilization of soil. This paper describes a number of methods that have been tried in the field to achieve an economical and effective pulverization method. It has been shown that an acceptable degree of pulverization can be attained when the soil is handled mechanically at a particular moisture range by using agricultural machinery.

●EXPANSIVE SOILS (1) occur in different parts of the world. One such soil, commonly termed black cotton soil, has similar characteristics and forms one of the major soil groups (2) of India, covering an area of about 500,000 km² (Fig. 1). The soil is predominantly montmorillonite (3, 4) having high base exchange capacity. It is characterized by high swelling on wetting and excessive shrinkage on drying. When swelling is restricted, it results in the development of swell pressure (3). On account of these peculiar properties, the black cotton soil presents serious problems in the construction of roads. Even at places where the conditions for the development of swell pressure do not exist, the roads still fail because of poor supporting power of the subgrade in wet condition.

It has been observed that the waviness at the road surface is mostly due to the working up of the soft subgrade soil into the crevices of the stone soling, thereby dislodging the soling stone from its original position. The sinking of the stone soling into the soft subgrade is a continuous process, and no amount of strengthening of the existing pavement at the surface would remedy this defect. It is, therefore, very necessary that a compacted layer of nonexpansive material having low voids be provided to prevent the movement of subgrade soil into the crevices of the stone soling. Such materials that could be considered suitable are light-textured soils, sands, or gravelly soils. In India, sandy soils or sands are not generally found in black cotton soil areas. At places, however, granular material mixed with soil fines, locally called moorum, occurs and a compacted layer of this laid over the subgrade before placing the stone soling has led to satisfactory results.

There are, however, still very large areas where none of these materials occurs and the only alternative is to improve the existing soil for use as a subbase between the subgrade and the stone soling. One of the known effective methods (4, 5) to improve the engineering properties of black cotton soil is the stabilization with lime. To achieve these requirements, it is necessary that the soil, which is generally in the form of hard clods, be brought to a reasonable degree of fineness to facilitate uniform mixing of lime with soil.

Figure 1. Black soil region of India.

Whereas light-textured soils are generally in a friable state when removed from the fields, it is not so in the case of expansive black cotton soil, which is very soft and sticky in the wet condition but very hard in the dry state.

OBJECT OF THE STUDY

The object of the present study, therefore, is to evolve a technique for an effective and economic pulverization of black cotton soil, which is a prerequisite for the uniform mixing of soil with lime and the subsequent development of strength.

Before taking up the field study, it was considered necessary to define the degree of fineness of soil. According to unpublished literature from the British Road Research Laboratory, the degree of fineness, which is also commonly termed as degree of pulverization, is determined from the formula

$$\frac{W_1 - W_2}{W_1 + W_3} \times 100$$

where

W_1 = total weight of the sample,
W_2 = weight of the sample retained on $\frac{3}{16}$-in. sieve, and
W_3 = weight of the sample passing $\frac{3}{16}$-in. sieve and retained on No. 8 sieve (British standard sieve or B.S.S.) 2 mm size.

It will appear from this formula that the fineness is not only controlled by the percentage of material passing the $\frac{3}{16}$-in. sieve but also by the fraction smaller than 2 mm.

For stabilization of light-textured soils with cement, about a 65 percent degree of pulverization (6), according to this formula, is considered suitable; but for hard clods of black cotton soil, such a high degree of pulverization may not be a practical possibility

TABLE 1

EFFECT OF DEGREE OF PULVERIZATION OF SOIL ON THE SOAKED
CBR WHEN TREATED WITH 3 PERCENT COMMERCIAL
HYDRATED LIME (PURITY 40 PERCENT) AND
COMPACTED TO 1.5 gm/cc DENSITY

Sample	Percent Passing			CBR Soaked (percent)	Moisture Absorption (percent)
	1-in. Sieve	$\frac{3}{16}$-in. Sieve	No. 8 Sieve (B.S.S.)		
1	100.0	0.0	0.0	2.4	27.5
2	100.0	50.0	15.0	14.2	26.3
3	100.0	100.0	30.0	14.3	26.9
4	100.0	100.0	100.0	14.7	25.3

Note: Soil characteristics are liquid limit = 75.3 percent; plasticity index = 34.7 percent; and fraction coarser than No. 200 sieve (U.S. sieve) = 5.0 percent.

(7), 8). It was, therefore, considered necessary to relax the limits of fraction finer than 2 mm from the calculations. Before finally accepting the modification, it was essential to know how the clods of black cotton soil pulverized to particles of varying sizes will affect the resultant strength in saturated condition when the soil is stabilized with lime. Toward this objective, a preliminary laboratory study was carried out with soil samples having varying clod sizes. To achieve a limited increase in strength, trials were made using a low concentration of lime.

In the laboratory trials, the soil having a varying degree of fineness was compacted at optimum moisture with 3 percent commercial lime of known purity. After curing the treated specimens for 10 days, these were tested for soaked CBR. The results obtained are given in Table 1.

It will be observed from the data given in Table 1 that if the soil to start with consists of at least 50 percent passing the $\frac{3}{16}$-in. sieve then the strength attained is practically the same, irrespective of the fraction passing the No. 8 sieve (B.S.S.). It may be due to the fact that the process of mixing lime with soil and subsequent compaction may have resulted in further improving the degree of pulverization.

PULVERIZATION BY MANUAL LABOR

It is a common experience that during the rainy season the black cotton soil is sticky and difficult to handle. The field trials on the pulverization of soil by manual labor were, therefore, restricted to the dry season; details are given in the following.

Crowbar and Pickax

The field trials for the pulverization of black cotton soil were carried out on Berasia-Sironj Road near Bhopal (central India) in 1964. After removing the top vegetation, the dry soil crust for a depth of about 8 in. was loosened with crowbars. The soil thus obtained was comprised mostly of 5- to 6-in. clods. These were broken with pickaxes or rammers but the output was very poor, which raised the cost to 20 rupees per 100 cu ft for getting soil of acceptable degree of pulverization, i.e., about 100 percent passing the 1-in. sieve and about 50 percent passing the $\frac{3}{16}$-in. sieve. With a view to economizing on cost, an attempt was made to use a country plow drawn by a bullock, instead of digging manually, but this did not work well on account of the soil being dry and hard.

Wetting and Drying of Soil Clods

It has been observed that, when black cotton soil shrinks in the process of drying, high stresses are produced that lead to the disintegration of soil at the surface. Advantage was taken of this phenomenon in the pulverization of the soil. In actual practice, the soil dug from the fields was stacked and water was sprinkled on the clods. In the process of drying, the shrinkage of the soil took place and led to its disintegration

at the surface. The process was frequently repeated to get more and more of the soil fines. It gave satisfactory results as almost 50 percent of the material passing the $^3/_{16}$-in. sieve could be obtained. This process would, however, require water within economic reach. Besides being slow, such operations will cost about 12 to 15 rupees per 100 cu ft.

PULVERIZATION BY MECHANICAL MEANS

Power Roller

The soil in the dry condition was dug from the adjoining fields, and clods were broken with pickaxes so as to reduce them to a size not bigger than 2 in. The soil clods were spread over a hard subgrade, and a power roller passed over them a number of times with frequent raking of the crushed material. It was found that about 8 passes of the roller and frequent raking of the rolled soil resulted in grading as follows:

Sieve No.	Percent Passing
1½ in.	100.0
1 in.	83.0
$^3/_8$ in.	80.0
$^3/_{16}$ in.	60.0

The cost of soil pulverized according to this method worked out to 10 to 12 rupees per 100 cu ft.

Heavy Agricultural Machinery

In the absence of a specially designed plant for the purpose, agricultural machinery available in the country was used. Field trials were, therefore, carried out with the following heavy agricultural machinery normally used for plowing the field and breaking clods (Fig. 2):

1. International Caterpillar tractor, 110 hp;
2. Moldboard plow consisting of 4 plowshares that can plow to about 15 in. in depth;
3. Disc plow consisting of 5 discs 28 in. in diameter with a working width of 10 ft;
and
4. Offset notched disc harrow consisting of 18 discs 22 in. in diameter arranged in 2 gangs with a working width of 10 ft.

A field trial was initiated at Sehore (central India) by using this machinery in April 1964 when the ground surface was hard and badly cracked because of a hot, dry summer. To start with, the moldboard plow with a working width of 6 ft was used. This could plow up to a depth of about 15 in., giving clods of varying sizes with a maximum of about 8 in. After the moldboard plow was used, the disc plow was operated on the excavated soil. This reduced the size of the big clods to about 4 in. in the process of slicing. The soil was further subjected to the action of the offset notched disc harrow to improve pulverization. It was observed that, even with 6 passes of the disc harrow, there were still many clods 4 in. in size that resisted pulverization and were therefore removed manually. The sieve analysis of the resultant soil is as follows:

Sieve No.	Percent Passing
1½ in.	100.0
1 in.	81.4
$^3/_8$ in.	57.6
$^3/_{16}$ in.	33.5

During this operation, it was observed that those clods that were dry and consequently hard resisted breaking up, whereas slightly wet clods could be pulverized easily. It was, therefore, inferred that the pulverization of black cotton soil with heavy agricultural machinery required the soil to exist within a certain range of

Moldboard plow
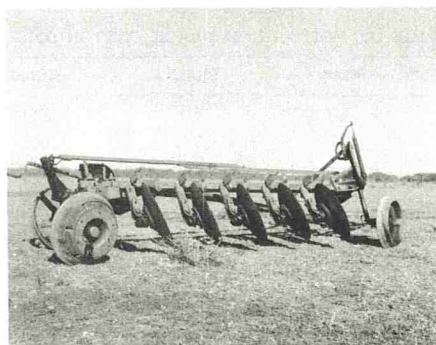
Disc plow

Offset notched disc harrow

Pulverized black cotton soil being carried by the laborers

Figure 2. Agricultural machinery.

moisture for effective pulverization. With a view to finding out the range of moisture that would facilitate pulverization, soil samples at various depths of the natural subgrade were taken and subjected to moisture tests. The results are as follows:

Depth, in.	Moisture, Percent
6	7.1
12	15.6
18	18.2

It can be inferred that the clods that could not be pulverized had a moisture content of about 7 percent and those that could be readily pulverized had a moisture content ranging between 15 and 18 percent. It can, therefore, be stated that it will be more economical to pulverize black cotton soil after the rainy season when uniform field moisture conditions are likely to prevail.

Experiments were also made on the pulverization of black cotton soil in a moist state. Before operating the machinery, the moisture distribution to a depth of 2 ft was checked at 3 different stretches on the Sehore-Bilquishganj Road (central India) where the experiments were carried out. The data are given in Table 2.

It will be observed that the moisture throughout the depth is very uniform except at the top where there is a slightly lower moisture content.

The field trials were carred out in December for a length of about 440 yards using the same machinery.

Depth, in.	Site 1	Site 2	Site 3
1	14.7	15.1	14.8
6	19.8	19.5	19.8
12	20.4	19.9	20.9
18	20.5	21.6	21.9
24	20.5	22.0	21.9

TABLE 3

SIEVE ANALYSIS OF BLACK COTTON SOIL
PULVERIZED IN THE MOIST STATE
WITH AGRICULTURAL MACHINERY

Sieve No. (B.S.S.)	Percent Passing		
	2 Passes	4 Passes	6 Passes
$1\frac{1}{2}$ in.	100.0	100.0	100.0
1 in.	82.6	88.3	89.6
$\frac{3}{8}$ in.	70.6	69.9	79.0
$\frac{3}{16}$ in.	44.8	49.6	55.8
No. 8	29.1	34.3	38.6
No. 36	5.2	6.8	8.2

It was observed that, after the soil had been dug out with the moldboard plow, the disc plow could conveniently cut down the big clods to a smaller size at this moisture. The operation was also smoother as compared to pulverization of the dry soil. The soil thus pulverized was further subjected to the action of the offset notched disc harrow, with a view to determining the minimum number of passes needed to achieve the required degree of pulverization. The soil was tested for sieve analysis after 2, 4, and 6 passes. The results are given in Table 3.

It will appear from the data given in Table 3 that the acceptable limits of pulverization, i.e., 50 percent passing the $\frac{3}{16}$-in. sieve, could be achieved with 6 passes of the offset harrow. It will be observed further that insofar as the upper limits are concerned the conditions are just satisfied. It will be found more economical to accept about a 10 percent fraction coarser than 1 in. than to make additional passes of the offset disc harrow to pulverize it further. It will also be noticed that with this type of machinery the moisture for effective degree of pulverization ranges between 15 and 20 percent. The cost of pulverization as worked out by this machinery is 1 rupee per 100 cu ft as given in Table 4.

Light Agricultural Machinery

As heavy agricultural machinery is not easily available at most of the sites, further field trials were carried out using light agricultural machinery, which is readily procurable. The trials were made at Sidhantam in Andhra Pradesh (south India) where the construction of $5\frac{1}{2}$ miles of the right approach road to Vasista Bridge, forming a part of the National Highway, was undertaken.

The light machinery consisted of the following:

1. Tractor, 50 hp;
2. Moldboard plow consisting of 3 plowshares;
3. Disc harrow consisting of 20 saucer-shaped discs 10 in. in diameter; and
4. Offset disc harrow consisting of 10 discs 20 in. in diameter arranged in two gangs.

The operation with this machinery was carried out, as in the case of heavy agricultural machinery, at varying moisture contents. It was found that effective pulverization could be economically achieved when the moisture content of the soil ranges between 10 and 22 percent, as against 15 and 20 percent in the case of the heavy machinery. It was further observed that it required about 6 passes of the disc harrow and about 10 passes of the offset harrow to achieve an acceptable degree of pulverization.

The cost of pulverization worked out to 2 rupees per 100 cu ft of loose soil as given in Table 5.

The higher cost of pulverization with light agricultural machinery as compared to heavy machinery resulted from the fact that the moldboard plow fitted with a light tractor could not plow more than 8 in. deep.

As a result of conducting a number of trials, it was found that, with the light machinery, it was possible to pulverize only about 8,000 cu ft of the soil in 8 hours as against 40,000 cu ft with heavy machinery during the same period. Although heavy

TABLE 4		TABLE 5	

COST ANALYSIS OF PULVERIZATION OF BLACK
COTTON SOIL WITH HEAVY AGRICULTURAL
MACHINERY

COST ANALYSIS OF PULVERIZATION OF BLACK
COTTON SOIL WITH LIGHT AGRICULTURAL
MACHINERY

Item	Amount	Item	Amount
Cubic feet of soil pulverized in 8 hours	40,000	Cubic feet of soil pulverized in 8 hours	8,000
Hire charges of the machinery at 45 rupees per hour	360.00	Hire charges of the machinery for 8 hours, rupees	120.00
Labor charges, rupees	20.00	Labor charges, rupees	20.00
Miscellaneous charges such as for tools and repairs, rupees	20.00	Miscellaneous charges such as for tools and repairs, rupees	20.00
Total rupees	400.00	Total rupees	160.00
Cost (in rupees) of pulverizing 100 cu ft $= \dfrac{100 \times 400}{40,000}$	1.00	Cost (in rupees) of pulverizing 100 cu ft $= \dfrac{160 \times 100}{8,000}$	2.00

machinery is more economical to use, it is not readily available in the market. Therefore, even though the cost of pulverization with light agricultural machinery is slightly more, it is still preferred on account of its easy procurement either from the market or on hire from the agriculturists. The additional cost of pulverization is not likely to have any significant effect on the overall cost of road construction.

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Changes in the Characteristics of Cement-Stabilized Soils by Addition of Excess Compaction Moisture

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The compaction moisture of cement-stabilized soils is usually specified as the optimum moisture content to obtain maximum density as determined by the standard Proctor test. Previous investigations have shown that in some instances maximum density may not correspond to maximum strength. If compaction of the soil-cement mix is delayed, the relationship between compaction moisture and the strength and density of the soil-cement also changes. This study investigates the relationship between compaction moisture content and the strength, density, and durability of cement-stabilized soils in which compaction is delayed after mixing to correspond to typical highway construction practices. Four types of soil suitable for cement stabilization were investigated. The compaction moisture content was varied from 4 percent below to 4 percent above the optimum moisture content obtained by standard Proctor tests with no delay between mixing and compaction. At each of the moisture contents, and at the optimum cement content, specimens were compacted 0, 2, 4, and 6 hours after mixing with no intermittent mixing. Specimens were prepared for unconfined compressive strength and durability tests. The results of this investigation show that the loss in strength and durability of soil-cement resulting from a delay in compaction can be significantly reduced in many instances by the addition of excess compaction moisture. The soils most benefited after a delay in compaction by excess moisture were the silty loams and sandy loams. Strength increases of 40 to 50 percent were achieved with these soils by the addition of 2 to 4 percent excess moisture when compaction was delayed. Cement-stabilized silty clay loams and silts compacted after delays showed little improvement in strength and durability with excess compaction moisture. Without delay in compaction, only the silty clay loams were significantly improved in strength and durability by the addition of excess compaction moisture. A study of the data has indicated that the amount of excess moisture required for maximum strength and durability depended on the soil type and the detention time between mixing and compaction. In granular soils the addition of excess moisture improved the strength and durability after delays in compaction. This improvement resulted from the improved lubrication of the soil aggregates and subsequent increase in dry density. With fine-grained soils excess moisture improved the properties of soil-cement mixes compacted without delay by increasing the amount of cement hydration.

•THE STANDARD PROCTOR METHOD is recommended to determine the compaction moisture requirements for soils stabilized with cement. Several investigators have found that this moisture content does not necessarily produce maximum density and

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maximum strength and durability. Felt (1) reported as early as 1955 that for maximum effectiveness portland cement and sand mixtures should be compacted slightly below the optimum compaction moisture content for maximum density and that silty and clayey soils stabilized with cement should be compacted 1 to 2 percent above the optimum.

Davidson et al. (2) also reported that the moisture contents for maximum strength are generally on the dry side of the optimum for predominantly sandy soils, and above the optimum moisture content for soils rich in clay. They postulated that the variation between optimum moisture content for maximum density and maximum strength was related to the particle sizes in the soil. They proposed that soils having a large surface area absorb much of the added water for lubrication so that insufficient water is available for hydration of the cement.

In the field, the time between the mixing of the soil and cement and the compaction of the soil-cement mixture is normally from 2 to 4 hours. Investigators studying the effects of delaying the compaction of soil-cement mixtures have reported large losses in the compressive strength, density, and durability if the delay in compaction exceeds 2 hours (3, 4, 5). They also found that the moisture content to obtain maximum density changes with delay in compaction. Data taken by West while working with sandy soils showed that after delay in compaction the moisture-density curves tended to change from the usual convex parabola to a concave parabola with the largest reduction in density occurring near the optimum moisture content.

The purpose of this study was to investigate the relationship between molding water content and the strength, density, and durability of cement-stabilized soils both with and without delay in compaction.

TESTING PROCEDURE

Ten natural soils considered suitable for cement stabilization were selected from various locations in Louisiana. The properties of these soils are given in Table 1. Each soil was stabilized with the optimum amount of cement as determined by the criteria developed by the Portland Cement Association based on freeze-thaw and wet-dry tests. Standard Proctor test ASTM D 558-57 was used to determine the optimum moisture content of the soil-cement mixture. Specimens were molded at the optimum moisture content and at 2 and 4 percent above and below the optimum.

Samples were not compacted at moisture contents higher than 4 percent above the optimum. Soils at higher moisture contents were not workable because they were above the plastic limit. Samples were molded according to ASTM D 1632-63 for unconfined compression tests and according to ASTM D 559-57 for wet-dry durability tests.

TABLE 1

SOIL PROPERTIES

Soil Identification	AASHO Group	Optimum Percent		Liquid Limit[c]	Plasticity Index[c]	Composition[d] (percent)		
		Cement[a]	Moisture[b]			Sand	Silt	Clay
Silty loam L-1	A-4	10	17.0	27	8	28	60	12
Silty loam L-2	A-4	10	17.0	28	8	7	79	14
Silty loam L-3	A-6	12	15.5	31	11	32	51	17
Silty clay loam L-4	A-4	10	17.5	28	9	26	53	21
Silty clay loam L-5	A-6	12	17.0	37	19	19	62	19
Silty clay loam L-6	A-6	12	20.0	38	14	28	51	21
Sandy loam L-7	A-2-4	7	15.0	25	7	71	16	13
Sandy loam L-8	A-2-4	7	10.5	18	1	67	21	12
Silt L-9	A-4	10	17.5	28	6	5	80	15
Silt L-10	A-6	12	19.0	36	11	0	87	13

[a] According to criteria established by the Portland Cement Association.
[b] Standard Proctor test, ASTM D 558-57.
[c] Atterberg limits, ASTM D 423-61T and ASTM D 424-5.
[d] Grain size analysis by ASTM D 422-63.

In order to study the effects of moisture content on the strength, density, and durability after a delay in compaction, samples were molded approximately 12 minutes after mixing had begun and at 2, 4, and 6 hours after initial mixing. All specimens prepared for strength studies were cured for 7 days in a 100 percent humidity chamber and then immersed in water for 4 hours prior to compression testing. Samples prepared for durability studies were tested according to ASTM D 599-57. All tests were run in triplicate, and over 1,200 samples were molded and tested during the course of this investigation.

At various detention times and moisture contents, portions of selected soil-cement mixtures were freeze-dried to prevent further hydration of the cement. Electron and optical microscopic studies and hydrometer and sieve analysis were conducted on the dried samples to study changes in particle shape and size. Isopropyl alcohol was used in the hydrometer and sieve analysis to prevent further cement hydration during the course of the experiment.

Chemical analysis and X-ray diffraction studies were also conducted to compare the extent of cement hydration at various moisture contents and detention times. The chemical analysis consisted of a spectrophotometric determination of the amounts of acid-soluble silica formed by the hydration of portland cement. Following the procedure developed by Ruff and Ho (6), the acid-soluble silica formed as the cement hydrate was extracted with 0.2N HCl during a half hour of vigorous shaking. The soil particles were removed from the liquid by centrifuging at 30,000 rpm for 5 minutes. Ammonium molybdate was then added to the solution and the absorption measured at 400 mu with a Beckman DU spectrophotometer.

The extent of cement hydration in soil-cement mixes at various moisture contents was also measured by X-ray diffraction. Powder specimens were X-rayed from 2 deg 2 theta to 40 deg 2 theta in a Philips Norelco diffractrometer using copper radiation. The reflections at 2.77 and 4.93 Å where chosen for study. The strong reflection at 2.77 Å

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                      ┌──────────────────────────┐
                      │   Preparation of Soil     │
                      └──────────────────────────┘
                                  │
                  ┌──────────────────────────────────┐
                  │ Classification of Soil - AASHO Groups │
                  └──────────────────────────────────┘
                                  │
                  ┌──────────────────────────────────┐
                  │ Determination of Optimum Moisture │
                  │        and Cement Content         │
                  └──────────────────────────────────┘
                                  │
                  ┌──────────────────────────────────┐
                  │ Mixing of Soil-Cement-Water 8 min │
                  │  with Counter-Current Bath Mixer  │
                  └──────────────────────────────────┘
                        │                      │
    ┌──────────────────────────┐      ┌──────────────────┐
    │ Compaction of specimens at 0, │  │ Freeze - Drying  │
    │ 2, and 4% above and below the │  └──────────────────┘
    │ optimum compaction moisture   │           │
    │ content, and at 0, 2, 4, and  │  ┌──────────────────────────┐
    │ 6 hr. delay between mixing    │  │ Extent of cement hydration by │
    │ and compaction for unconfined │  │ chemical and x-ray diffraction │
    │ compressive strength and      │  │ analysis                  │
    │ durability tests              │  └──────────────────────────┘
    └──────────────────────────┘               │
                                      ┌──────────────────────────┐
                                      │ Changes in particle size and │
                                      │ shape from electron and optical │
                                      │ microscopic studies and hydro- │
                                      │ meter and sieve analysis  │
                                      └──────────────────────────┘
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Figure 1. Flow diagram of experimental procedures.

L-5, and L-6 are shown in Figure 3. The silty clay loams developed maximum strength at higher than optimum moisture content even for samples compacted without delay. With delay in compaction, the strength generally increased and then decreased as the molding water content was increased above the optimum.

After a delay in compaction, the density consistently decreased with increasing moisture content.

Silty clay loams compacted without delay showed improved durability at moisture contents higher than the optimum. The durability of samples compacted after a delay was poor regardless of the molding moisture content.

Sandy Loam

The sandy loam soils L-7 and L-8 had varied results as shown in Figure 4. The strength of soil L-8 at optimum moisture content was reduced over 50 percent by a delay in compaction of 2 hours or more. Varying the percentage of compaction moisture did not improve the strength a significant amount. On the other hand, increasing the percentage of molding water 2 percent above the optimum reduced the loss of strength after a 2-hour delay of soil L-7 from 23 to 3 percent.

The moisture-density curves of the sandy loam soils were concave after a delay in compaction instead of the normal convex shape. Maximum densities were obtained above the optimum moisture content with no delay.

With no delay in compaction, maximum durability was achieved at optimum water content. The durability of samples compacted after a delay was greatly improved by the addition of excess compaction moisture.

Silt

The variations of strength, density, and durability with moisture content for silts L-9 and L-10 are shown in Figure 5. The optimum moisture content for maximum strength and density did not change significantly with a delay in compaction.

These 2 soils differed appreciably in durability after delay in compaction. In general, the durability improved with increasing compaction moisture. Soil L-9 proved to be a very durable material for soil-cement stabilization even after delays in compaction of up to 6 hours. Soil L-10, although giving higher initial strength at 1-hour delay, had lower durability than L-9 at all moisture contents.

General

The results obtained for the 10 soils show that, when compaction of the soil-cement mix is delayed, maximum strength and durability in most instances are not obtained at the optimum moisture content for maximum density as determined with no delay but developed at moisture contents above the optimum. Even with no delay in compaction, the silty clay loams developed maximum strength and durability at higher than optimum moisture contents. A study of the data indicates that the increase in strength and durability with excess compaction moisture is due to a combination of increased dry density and improved cement hydration.

The reaction of cement with soil in the soil-cement matrix is very vigorous and is a function of time and moisture content. If compaction is delayed after mixing, cementation of the loose soil grains into larger aggregates takes place. This conglomeration effect increases the resistance of the soil to compaction so that the density obtained at a given moisture content and compactive effort is reduced.

As mentioned earlier, delay in compaction also tends to increase the moisture required to obtain maximum density. The necessary increase in compaction moisture with delay in compaction seems unusual because large soil particles normally require less water for lubrication than small particles. However, consideration of the shape of the soil aggregates explains why more moisture is required for lubrication. Figure 6 shows the configuration of the soil aggregates as seen with an optical microscope. The extremely irregular shapes of the aggregates increase the resistance to compaction by mechanical interlocking. If sufficient water is added to form a continous film around
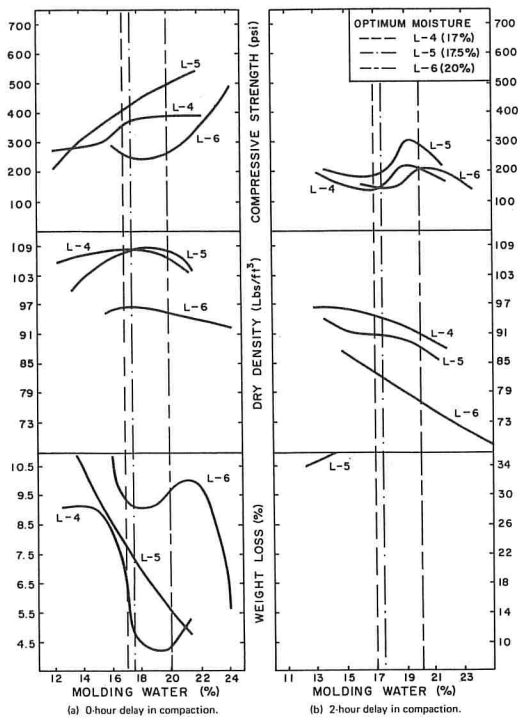
Figure 3. Effect of compaction moisture on silty clay loam soil-cement mixes at various detention times.
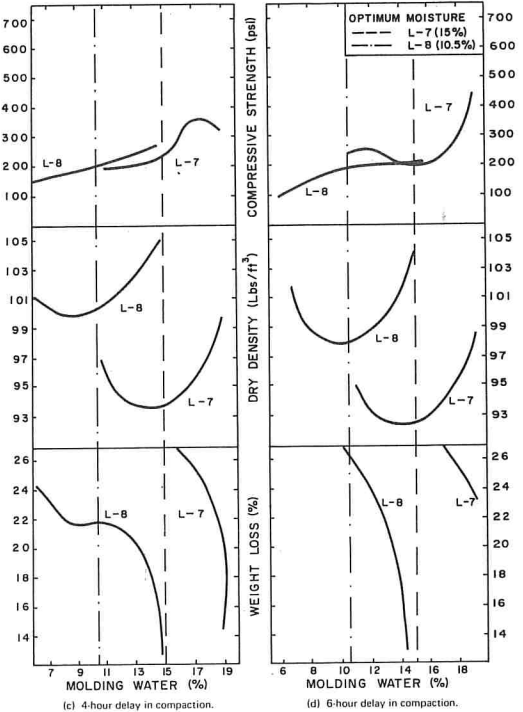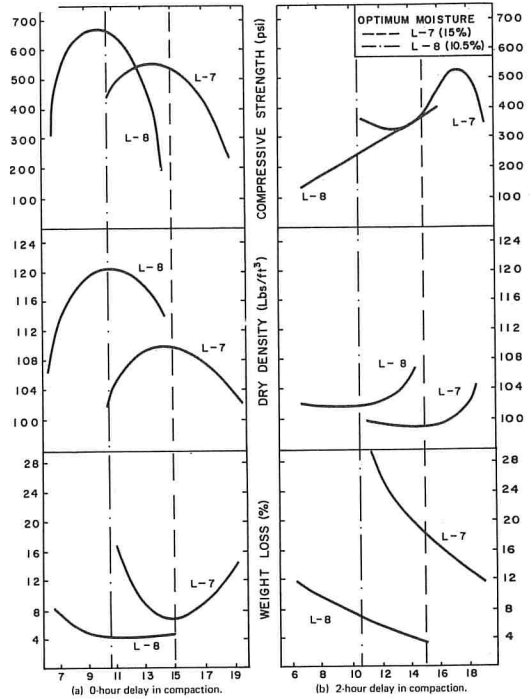
Figure 4. Effect of compaction moisture on sandy loam soil-cement mixes at various detention times.

Figure 5. Effect of compaction moisture on silty soil-cement mixes at various detention times.
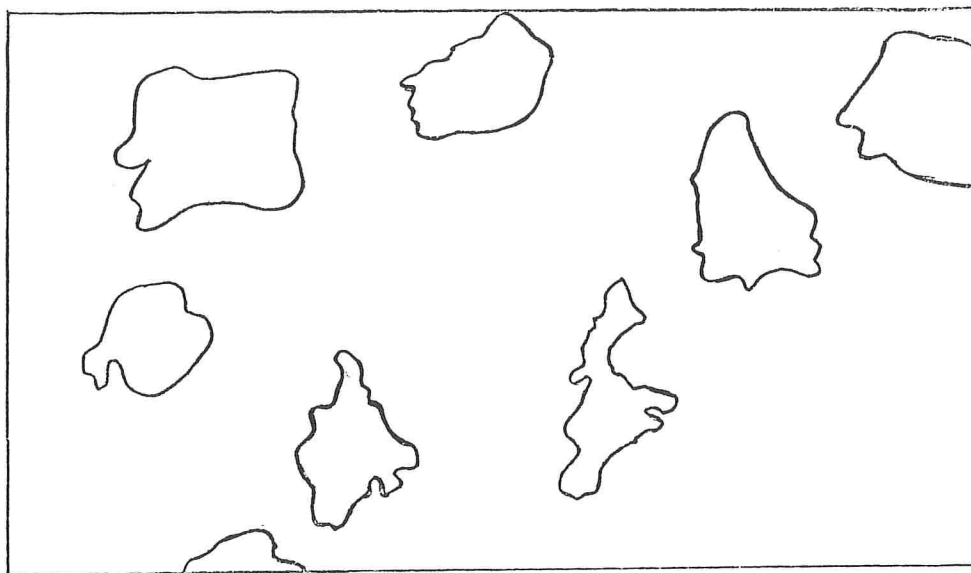
(a) 0-hour after mixing (magnification of 750x)
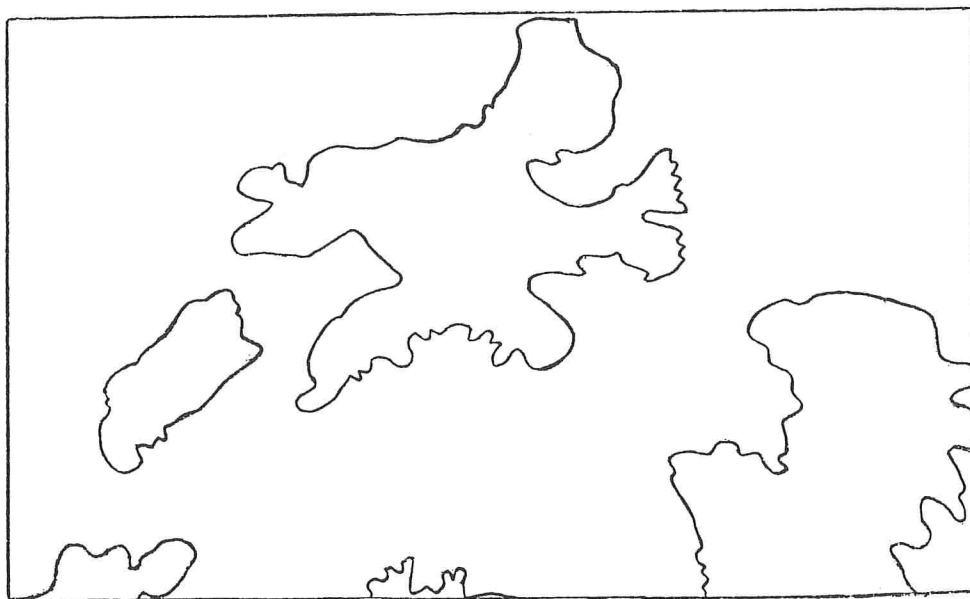
(b) 6-hours after mixing (magnification of 750x)

Figure 6. Effect of delay in compaction on the size and shape of soil particles.

the aggregates to prevent excessive interlocking, the resistance to compaction will be lower and the dry density will be increased. A schematic representation of this effect is shown in Figure 7.

The extent of soil particle aggregation with detention time was measured by a hydrometer and sieve analysis of the soil-cement mixture. Typical data for a silty clay loam are shown in Figure 8. These data indicate that considerable aggregation does occur
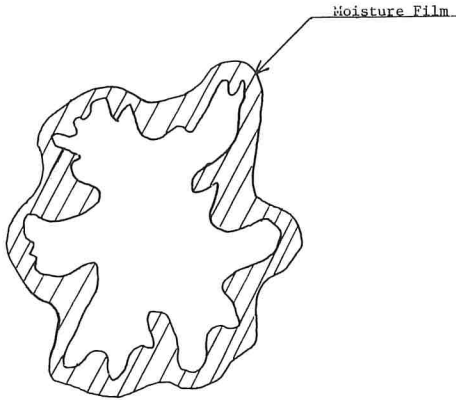
Figure 7. Hypothesis of reduction in mechanical interlocking at high moisture contents.

TABLE 2

EFFECT OF PERCENTAGE OF COMPACTION MOISTURE ON THE EXTENT OF CEMENT HYDRATION IN SILTY CLAY LOAMS

X-Ray Diffraction of Specimens Cured for 7 Days

Compaction Moisture (percent)	Tricalcium Silicate Line (2.77 Å)	Calcium Hydroxide Line (4.93 Å)	Quartz Line (2.45 Å)
10	15.9	3.2	100
12	7.0	3.0	100
14	6.0	4.0	100
16	11.0	2.0	100
18	8.0	7.0	100

Chemical Analysis of Acid-Soluble Silica

Compaction Moisture (percent)	Acid-Soluble Silica (g/100 g soil)		
	6-Hour Curing	1-Day Curing	7-Day Curing
10.0	0.68	0.86	1.04
12.5	—	1.03	1.08
15.0	0.74	1.02	1.07
17.5	0.74	1.06	1.05
20.0	—	1.11	0.98
22.5	—	0.95	0.96
25.0	—	1.31	1.20

with a delay in compaction. A 6-hour delay in compaction reduced the weight of particles less than 100 microns by 23 percent.

The increase in strength and durability at higher than optimum moisture contents after delay in compaction of the silty loam and the sandy loam soils can be attributed to the increase in dry density. The danger of directly relating the dry density and the strength and durability is readily apparent in Figure 4 (c and d). After a delay in

Figure 8. Effect of delay in compaction on soil particle size.

TABLE 3

COMPACTION MOISTURE FOR OPTIMUM SOIL-CEMENT PROPERTIES

Soil Identification	Composition (percent)			Compaction Moisture for Maximum Strength and Durability [a]			
	Sand	Silt	Clay	0-Hour Delay	2-Hour Delay	4-Hour Delay	6-Hour Delay
L-1	28	60	12	17.0	21.0[+]	21.0	21.0
L-2	7	79	14	17.0	21.0[+]	21.0[+]	21.0[+]
L-3	32	51	17	15.5	15.5	17.5	17.5
L-4	26	53	21	19.0	19.0	19.0	19.0
L-5	19	62	19	21.5[+]	19.5	19.5	19.5
L-6	28	51	21	24.0[+]	20.0	20.0	22.0
L-7	71	16	13	15.0	17.0	17.0	19.0[+]
L-8	67	21	12	10.5	14.5[+]	14.5[+]	14.5[+]
L-9	5	80	15	17.5	19.5	21.5	19.0
L-10	0	87	13	19.0	23.0[+]	23.0[+]	19.0

[a] + after moisture content denotes continuous increase in strength and durability with increasing moisture content.

compaction, the density of the sandy loam soils is significantly increased at moisture contents lower than the optimum moisture content obtained at 0-hour delay. However, at the low moisture contents, the strength and particularly the durability are much less than at higher moisture contents. The higher densities at low moisture content result from the small amount of cement hydration and subsequent small amount of soil aggregation.

The explanation for the behavior of the silty clay loams is not the same as for the silty loams and sandy loams. The strength of the silty clay loams continued to increase with an increase in the moisture content up to 4 percent above the optimum with no delay in compaction. When compaction was delayed, the strength of the silty clay loam soils tended to increase with increasing molding water content even though the density consistently decreased.

One explanation for this behavior as suggested by Davidson et al. (2) is that the moisture content gave maximum density and much of the water added for lubrication is absorbed by the clay particles in the soil, resulting in insufficient water for complete hydration of the cement. Additional compaction moisture above the optimum increases the amount of cement hydration and hence the strength, even though the density is reduced.

To determine if the addition of excess moisture above the amount necessary for maximum density did in fact increase the amount of cement hydration, silty clay loam soil-cement mixtures were compacted at various moisture contents. X-ray diffraction studies and spectrophotometric analysis were conducted as outlined earlier.

The results of these studies are given in Table 2. The results, although inconclusive, indicate a general trend to increased cement hydration in the silty clay loams compacted at moisture contents above that required for maximum density.

Table 3 gives the data collected during this investigation. The results indicate that the amount of excess compaction moisture required for maximum strength and durability depends on the soil type and the detention time between mixing and compaction. If the detention time was 2 to 6 hours, the strength and durability of the more granular soils were significantly improved by the addition of 2 to 4 percent excess compaction moisture. With no delay in compaction, only the fine-grained silty clay loam showed improvement with excess compaction moisture.

CONCLUSIONS

The following conclusions were drawn from the results of this investigation.

1. The moisture content for maximum strength and durability is not necessarily equal to the moisture content that gives maximum density.

2. The rate and direction of change in moisture content, which gives maximum strength and durability after a delay in compaction, are often not the same as the rate and direction of change of the moisture content required for maximum density.

3. The amount of excess compaction moisture required for maximum strength and durability depends on the soil type and the detention time between mixing and compaction. Two to 4 percent excess compaction moisture significantly improves the strength and durability if (a) the detention time is greater than 2 hours and the soils is granular and (b) the detention time is less than 2 hours and the soil is fine-grained.

4. After delays in compaction, the strength and durability of silty loams and sandy loams are significantly improved at moisture contents 2 to 4 percent above the optimum determined at 0-hour delay. The change in moisture content for maximum strength and durability with delay in compaction is due to aggregation and mechanical interlocking of soil particles. The addition of excess moisture lubricates the soil aggregates and increases the density, strength, and durability.

5. The moisture content giving maximum strength and durability with silty clay loams is 4 percent above the optimum with no delay in compaction, and 0 to 2 percent above the optimum with delay in compaction. The increase in strength at moisture contents up to 4 percent above optimum when compaction is not delayed appears to be due to more efficient cement hydration.

6. The moisture-density relationships for silts change little with delay in compaction. However, improvement in the durability of silts is achieved at moisture contents 2 to 4 percent above the moisture content for maximum density with no delay in compaction. Little improvement in the strength is obtained at higher moisture contents.

7. It is recommended that, if delays in compaction of 2 to 6 hours are expected, the compaction moisture of cement-stabilized silty loams, silty clay loams, sandy loams, and silts should be 2 to 4 percent above the amount required for maximum density as determined by Proctor test performed with 0-hour delay. The compaction moisture content of silty clay loams should be 3 to 4 percent above the optimum moisture content even if no delay in compaction is expected.

8. The use of additional moisture does not completely counteract the detrimental effects of delay in compaction; however, it will significantly improve the properties of compacted soil-cement mixtures with very little extra cost. Additional studies are being conducted to determine methods of completely overcoming the deficiencies in soil-cement as a result of delay in compaction. Until methods of eliminating the adverse effects of delayed compaction are perfected, time restrictions between mixing and compaction should be strictly enforced.

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Reactions and Strength Development in Portland Cement-Soil Mixtures

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The ultimate goal of this study was to gain insight into the reactions
occurring in portland cement-soil mixtures. Soils of the Frederick,
Iredell, and Lloyd series commonly found in Virginia were investi-
gated. The effect of their chemical, mineralogical, and engineering
properties and soil weathering stage on such processes as the deteri-
oration of the soil constituents, the production and consumption of
calcium hydroxide, and strength development of the mixtures is dis-
cussed. Investigative techniques used included X-ray diffraction and
microscopic analyses, and chemical analyses of water (for Ca) and
HCl (for Si, Al, and Fe) leachates of the mixtures. The Frederick
soil constitutents did not appreciably react with the cement, although
some of the clays in the soil were slightly deteriorated after cement
treatment. The clays and probably other constituents in the B- and
C-horizons of the Iredell and Lloyd soils suffered deterioration after
cement treatment, as indicated by the release of large amounts of
silica and alumina and by the diminution of mineral X-ray peaks.
When the major clay mineral in the soils was considered, the soils
suffered deterioration after cement treatment from greatest to least
in the following order: montmorillonitic soils, kaolinitic soils, illitic
soils. The instability of the C-horizon Iredell soil constituents in a
cement system appeared related to mineralogy. The minerals were
not greatly weathered and were susceptible to the harsh conditions of
an alkaline environment. The greatest quantities of calcium hydroxide
were consumed with kaolinite as the dominant clay mineral. It ap-
pears that reaction of calcium hydroxide with the soil constituents was
directly proportional, and the development of strength was inversely
proportional, to the magnitude of the clay-size fraction.

●THIS RESEARCH is an outgrowth of a previous study conducted by David F. Noble in
1967 on the reactions occurring in mixtures of portland cement and relatively pure
clays. It was decided to extend the work of Noble to the consideration of clays in their
natural environment of soils. As an ultimate goal it was hoped to gain insight into the
reactions occurring in portland cement-soil mixtures by studying the reaction products
of the mixtures; the rates of reaction of the mixtures; the deterioration or lack of de-
terioration of clay minerals in the mixtures; and the development of strength in the
mixtures.

MATERIALS INVESTIGATED

Cement

Chemical and physical analyses of the type I cement used in this study are given in
Table 1.

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TABLE 1

CHEMICAL AND PHYSICAL ANALYSES OF
CEMENT UTILIZED

Item	Amount	Item	Amount
Oxide Analysis, percent		Compounds, percent	
SiO_2	21.20	C_3S	52.0
Al_2O_3	5.90	C_2S	21.6
Fe_2O_3	2.34	C_3A	11.68
CaO	64.44	C_4AF	7.11
MgO	2.83	$CaSO_4$	3.71
SO_3	2.18		
Ign. loss	0.68	Fineness	
Na_2O	0.12	Percent passing	
K_2O	0.73	No. 325 mesh	91.8
Na_2O equiv.	0.60	Blaine, cm^2/gm	3,396
Free CaO	1.00		

Note: Cement and analyses courtesy of Standard Lime and Cement Division of Martin-Marietta.

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TABLE 2

SOIL SAMPLE DESIGNATIONS

Soil Series	Sample	Depth Sampled (ft)	Soil Horizon
Frederick	F-1	0 to 1.9	A
	F-2	1.9 to 3.8	B
	F-4	5.6 to 7.5	C
	F-8	13.1 to 15.0	C
	F-12	20.6 to 22.5	C
Iredell	I-1	0.7 to 1.3	A_2
	I-2	1.3 to 2.0	B
	I-5	3.3 to 4.0	C
Lloyd	L-1	0.5 to 2.2	B_1
	L-3	4.3 to 6.4	B_2
	L-5	8.5 to 10.6	C
	L-9	16.9 to 19.0	C

Soils

The 3 types of soil chosen for this study were (a) a limestone-derived silty clay of the Frederick soil series that is characterized by its yellowish-red to brownish-red color and fine-grain size (10); (b) a basic intrusive residuum of the Iredell soil series that is characterized by the shallow profile, dark color, poor internal drainage, and extremely plastic clay-pan horizon of the soil (14); and (c) a resilient micaceous soil of the Lloyd series that is characterized by its thick, heavy-textured subsoil, good drainage, high mica content, and deeply weathered profile (4). These types were chosen because they are residual, and thereby allow some correlation between parent material and derived soil.

The soils were sampled from fresh road cuts or sampling pits. Twelve specimens, each representative of a specific varied horizon of the soils, were selected for detailed study, although the entire profile of the soils was originally sampled. The properties of these selected samples are given in Tables 2 through 6. The properties were determined using standard soils, mineralogical, and chemical laboratory techniques.

TABLE 3

MINERALOGY OF THE SOILS

Sample	Sand Fraction[a]	Clay Fraction[b]
F-1	Fine-grained quartz, limonite, ferrugineous quartz aggregates	Quartz, illite, mixed layer clay, kaolinite, montmorillonite, goethite
F-2	Like F-1	Mixed layer clay, quartz, illite, kaolinite
F-4	Like F-1 except for a few chert fragments	Illite, quartz, mixed layer clay, kaolinite
F-8	Like F-4	Quartz, illite, kaolinite, mixed layer clay
F-12	Like F-4 except for a slight increase in ferrugineous material	Quartz, illite, kaolinite, mixed layer clay, montmorillonite, goethite
I-1	Quartz, limonite, chlorite, talc, hornblende, feldspar, biotite, ferrugineous quartz aggregates	Vermiculite, quartz, illite, chlorite, kaolinite
I-2	Biotite, chlorite, quartz, limonite, hornblende, feldspar, epidote	Illite, vermiculite, quartz, chlorite, kaolinite
I-5	Chlorite, biotite, hornblende, feldspar, quartz, many gabbroic fragments	Montmorillonite, illite, vermiculite, quartz, chlorite, kaolinite
L-1	Mica, quartz, feldspar, limonite	Kaolinite, illite, mixed layer clay, quartz
L-3	Mica, limonite, quartz, feldspar	Like L-1
L-5	Mica, limonite, quartz	Kaolinite, illite, quartz, mixed layer clay, montmorillonite
L-9	Quartz, mica, feldspar, several gneiss fragments	Kaolinite, illite, mixed layer clay, quartz, montmorillonite

[a]Minerals listed in order of decreasing abundance.   [b]Minerals listed in order of decreasing X-ray intensity.

TABLE 4

CHEMICAL ANALYSES OF THE SOILS, PERCENT

Sample	SiO$_2$	Al$_2$O$_3$	Fe$_2$O$_3$	CaO	MgO	Na$_2$O	K$_2$O	Ign. Loss	Total
F-1	74.50	12.07	5.27	0.14	2.10	1.74	1.95	4.28	102.05
F-2	68.00	15.43	6.68	0.26	1.85	0.16	1.96	5.70	100.04
F-4	69.33	14.38	6.38	0.00	2.06	1.01	2.01	5.48	100.65
F-8	67.50	14.47	5.98	0.10	2.31	1.55	2.43	5.63	99.97
F-12	72.00	12.98	5.80	0.06	1.76	1.88	2.56	4.98	102.02
I-1	54.40	10.40	23.89	1.43	0.00	2.60	0.67	6.65	99.94
I-2	49.92	21.33	14.93	1.88	1.34	2.06	0.68	9.31	101.45
I-5	45.89	17.07	10.67	7.90	10.09	1.61	1.55	3.64	98.42
L-1	39.36	30.55	16.33	0.28	0.00	0.34	1.53	11.75	100.14
L-3	43.67	25.22	18.44	0.00	0.79	0.58	2.41	10.03	101.14
L-5	42.03	23.43	18.46	0.14	1.28	1.16	3.48	8.65	98.63
L-9	60.43	19.05	6.36	0.22	0.00	2.13	5.30	6.41	98.12

TABLE 5

GRAIN SIZE ANALYSES OF THE SOILS

Sample	Percentage by Size Ranges in Microns									
	>2,000	2,000-420	420-62	62-31	31-16	16-8	8-4	4-2	2-1	<1
F-1	0.23	0.49	3.31	11.58	12.33	12.62	9.54	7.64	5.82	36.44
F-2	0.95	1.47	4.67	6.27	7.49	10.62	8.89	8.05	6.35	45.24
F-4	0.87	1.02	4.26	8.68	10.43	8.93	7.42	7.36	5.19	45.84
F-8	0.68	1.06	4.60	10.35	9.11	10.51	10.15	13.26	7.97	32.31
F-12	0.62	1.96	3.22	9.90	7.96	10.25	10.15	10.15	6.78	39.01
I-1	23.58	24.81	8.94	4.15	3.07	7.29	5.61	4.89	3.92	13.74
I-2	2.71	7.01	6.78	3.60	2.26	4.97	3.29	3.37	4.51	61.50
I-5	13.70	29.71	32.61	7.26	2.52	2.72	1.50	1.19	0.80	7.99
L-1	0.42	0.89	19.03	4.53	4.71	5.30	4.06	2.47	3.02	55.57
L-3	0.77	0.91	43.50	5.66	8.15	6.90	5.12	2.89	1.66	24.44
L-5	0.36	1.80	53.03	10.03	10.86	6.89	4.21	2.39	1.77	8.66
L-9	1.43	7.35	45.71	17.35	8.54	7.27	3.52	1.97	1.21	5.65

TABLE 6

ENGINEERING PROPERTIES OF THE SOILS

Sample	Liquid Limit (percent)	Plastic Limit (percent)	Plasticity Index (percent)	Optimum Moisture Content (percent)	Maximum Dry Density (lb/cu ft)	Engineering Classification
F-1	38	23	15	21.0	102.4	—
F-2	47	30	17	24.8	97.3	A-7-5 (12)
F-4	46	28	18	26.3	96.1	—
F-8	43	27	16	24.4	98.4	A-7-5 (11)
F-12	42	26	16	23.7	99.1	—
I-1	32	22	10	15.5	121.8	—
I-2	84	32	52	29.5	91.0	A-7-5 (20)
I-5	35	24	11	14.1	124.2	A-6 (8)
L-1	51	—	0	26.6	95.2	A-5 (10)
L-3	—	—	0	27.5	92.4	—
L-5	—	—	0	26.3	92.9	—
L-9	—	—	0	21.6	96.8	A-4 (0)

EXPERIMENTAL PROCEDURES

Leachates Made With H₂O and HCl

A more complete description of the procedures than that which follows is presented by Plaster (13).

As a means of investigating the chemistry of soil-cement reactions, the minus No. 230 sieve fraction of the soils was dry mixed with 40 percent portland cement by weight. These mixtures were then combined with distilled water at a solids-to-liquid ratio of 1 to 1 (by weight) and stored in plastic vials at room temperature for curing periods of 2, 21, and 56 days.

In order to determine what chemical interplay had occurred between the soil and cement, it was decided that a series of leaches made with distilled water and acid solutions might provide some insight into the reactions. After the distilled water leach, the pH was taken and the suspended material then removed by centrifugation. The distilled water leachate was set aside for calcium oxide analysis. Another sample of the soil-cement was leached with 200 ml of 1.0 N HCl. The acid leachate was set aside for analyses of silica, alumina, and iron oxide.

Calcium oxide was determined by titration with EDTA as described by Davis (6). Silica, alumina, and iron oxide were determined by absorption spectrophotometry (6).

X-Ray Studies

Randomly oriented powder specimens were X-rayed through an arc from 2 deg 2 theta to at least 45 deg 2 theta.

Microscopic Investigations

Because a study of the fabric and physical bond of soil-cement was considered of value, thin sections were made of representative specimens prepared as previously described. The curing periods were varied because of the convenient time for making the thin sections. The curing periods of the thin-section specimens were as follows:

Sample	Curing Period (days)
F-2	125
F-8	150
I-1	97
I-2	97
I-5	97
L-1	95
L-9	95

Thin discs were cut from the centers of the vials and impregnated under vacuum with Permount, a synthetic resin mixed with 40 percent toluene. After the discs were ground down to approximately 30 microns, the thin sections were studied under the petrographic microscope.

Strength Studies

In an effort to relate unconfined compressive strength gain to soil-cement reactions, 6 Harvard-size specimens of each soil sample and 6 more containing 10 percent portland cement were molded at optimum moisture content and maximum density. After curing for 7 days in a moisture room at 23 C, the specimens were tested in a Soiltest Versa-tester, Model 30M, stress-strain apparatus.

RESULTS AND DISCUSSION

Leachings of Calcium Oxide Made With Distilled Water

Water leaches, as stated earlier, were conducted at 2- and 56-day curing periods in order to determine the amount of easily soluble calcium in the various soil-cement
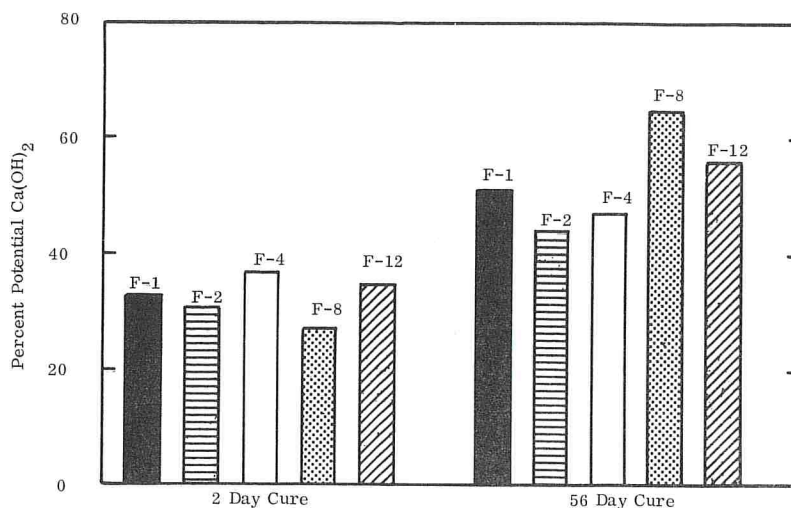
Figure 1. Percentage of potential Ca(OH)$_2$ leached from the Frederick soil-cement specimens.

mixtures. The mixtures, as shown by X-ray analysis, were either noncarbonated or only very slightly carbonated.

Calcium in hydrating cement is easily leached from only one source—calcium hydroxide, either crystalline or noncrystalline (11). It was therefore hoped that the quantity of calcium leached might serve as a measure of the amount of calcium hydroxide in the soil-cement specimens. This measure, in turn, when compared with other data, might serve as a guide to the extent of hydration of the samples and might offer some insight into any reactions occurring between the soil and portland cement.

Throughout the following discussion calcium data, given as a measure of calcium hydroxide production, are often referred to as a percentage of potential calcium hydroxide. This percentage of potential calcium hydroxide is based on calcium hydroxide being 25 percent of fully hydrated cement (3). The reader is reminded, however, that at neither the 2- nor 56-day curing periods would the cement be fully hydrated (5). Therefore, percentages of potential calcium hydroxide will always be less than 100 percent.

Frederick Soil-Cement Specimens—Although there were A-, B-, and C-horizons present in the Frederick soil sampled, physical and chemical properties did not vary greatly down the soil profile. Therefore, it was not surprising that, as is shown in Figure 1, the amount of potential calcium hydroxide leached from the 5 different samples was remarkably similar for the same curing periods.

The amounts of calcium leached by water after each curing period seem to indicate that cement hydration and the formation of calcium hydroxide were proceeding at a normal rate (15). The increase in amount of calcium leached from the 2-day curing period to the 56-day curing period indicates that calcium hydroxide was being produced at a rate greater than its consumption. Thus it is implied that no significant reaction has occurred between the soil constituents and the cement and that the soil materials have not restricted the hydration of the cement.

The data given in Table 7 attempt to show the correlation of the difference in percentage of potential calcium hydroxide leached by water from the 2-day and 56-day cured specimens to the mineralogy and magnitude of the clay-size fraction. As the data given in Table 3 show, the gross mineralogical composition of the Frederick clay-size fraction was relatively constant, although traces of montmorillonite and goethite were in F-1 and F-12. However, the relative quantities of the minerals varied slightly. With quartz and illite the dominant clay-size minerals in F-8, the greatest increase in leached calcium

hydroxide occurred. The principal varia-
tion in F-1 and F-12 as compared with F-8
was the presence of montmorillonite, and
the difference in leached calcium hydrox-
ide decreased. Without montmorillonite
but with the relative abundance of quartz
decreased and the total clay-size fraction
increased, as in F-2 and F-4, the calcium
hydroxide leached further decreased. It
is apparent that even extremely fine-
grained quartz neither impeded the forma-
mation nor contributed to the consumption
of calcium hydroxide. However, with a
significant increase in the relatively inac-
tive illite as well as the mixed-layer clay,
or with the appearance of montmorillonite,
either the production of calcium hydroxide
is hindered or some calcium hydroxide is
consumed.

The pH data on the Frederick soil-
cement specimens do not seem to indicate
anything in particular other than that there

TABLE 7

LEACHED $Ca(OH)_2$ AND CLAY SIZE DATA

Sample[a]	Clay Size Mineralogy[b]	Percent Clay of Material Passing No. 230 Sieve	Difference in Leached $Ca(OH)_2$ 56-days minus 2-days, percent
F-8	Q, I, K, ML	45.0	+38
F-1	Q, I, ML, K, M, G	44.2	+18
F-12	Q, I, K, ML, M, G	49.5	+21
F-2	ML, Q, I, K	55.5	+13
F-4	I, Q, ML, K	55.4	+10
I-5	M, I, V, Q, Ch, K	34.8	-6
I-1	V, Q, I, Ch, K	42.4	+5
I-2	I, V, Q, Ch, K	79.5	-1
L-9	K, I, ML, Q, M	17.2	-3
L-5	K, I, Q, ML, M	20.0	-6
L-3	K, I, ML, Q	48.2	-15.5
L-1	K, I, ML, Q	73.7	-31.0

[a]Listed in order of increasing clay percentage.

[b]Q = quartz; I = illite; K = kaolinite; ML = mixed layer; M = montmoril-lonite; G = goethite; V = vermiculite; and Ch = chlorite.

was little disparity in pH among the samples. The highest pH value was 11.61 (F-2, 56-day cure) and the lowest pH value was 11.12 (F-8, 2-day cure). These data again point up the uniformity of the Frederick samples in their reaction with cement.

There was a direct relationship between the pH of the Frederick soil-cement speci-mens and the amount of calcium leached. Thus, it appears that the amount of calcium hydroxide present in the specimens determines the alkalinity of the soil-cement system.

Iredell Soil-Cement Specimens—The Iredell soil, unlike the Frederick, displays a great deal of variation in properties with profile depth. As a result, samples from dif-ferent horizons of the soil reacted somewhat differently to cement treatment.

The leached A-horizon of the Iredell soil (sample I-1), as shown in Figure 2, yielded approximately 10 percent more calcium than the B- and C-horizons of the soil, and there was a slight increase from the 2-day to the 56-day cure in calcium leached. The specimens containing soil from the B- and C-horizons not only yielded less of the po-tential calcium hydroxide at both curing periods but also showed decreases from the 2-day to the 56-day cure in the amount of calcium leached. The factors influencing this

Figure 2. Percentage of potential $Ca(OH)_2$ leached from the Iredell soil-cement specimens.

disparity between the leached A-horizon and the B- and C-horizons concerning reactions to cement treatment will be discussed in the section on acid leaches.

The calcium value for the 2-day cure of the Iredell leached A-horizon soil-cement specimen very closely resembles those of the Frederick specimen, although all 3 Iredell specimens yielded much less calcium than the Frederick at 56 days. Apparently, in the leached horizon specimen of the Iredell, calcium hydroxide production was slowed after 2 days of curing, or calcium hydroxide was consumed in some manner other than in normal cement hydration processes. One possibility is that reaction of the cement with soil materials may have occurred, causing deletion of calcium hydroxide.

Calcium data on the specimens containing soil from the B-horizon (I-2) and the C-horizon (I-5) are remarkably similar, as is shown in Figure 2. This similarity is surprising because sample I-2 was a clay-pan soil and sample I-5 was a sandy saprolite. The data on both samples indicate that the presence of soil constituents either restricted the production of calcium hydroxide or caused rapid consumption of calcium hydroxide. Because the C-horizon of the Iredell soil is coarse-grained, it would not be expected that this soil would react to cement treatment in the same manner as the clayey B-horizon. It should be remembered, however, that only material passing the No. 230 sieve was used in the leachate studies. Of the minus No. 230 material, approximately 35 percent of the C-horizon soil was clay sized (< 2 microns). This high percentage of clay, in combination with other factors such as mineralogy, may account for the similar behavior of the B- and C-horizon soil-cement samples.

Data on pH's of the Iredell specimens were lower than those of the Frederick specimens. The values ranged from 10.21 (I-2, 56-day cure) to 11.30 (I-1, 56-day cure). Like the Frederick samples, the pH values roughly paralleled the amounts of calcium leached.

Lloyd Soil-Cement Specimens—Calcium data on the specimens containing Lloyd soil were neither consistently similar as in the Frederick specimens nor predictably dissimilar as in the Iredell specimens. As shown in Figure 3 the amount of potential calcium hydroxide leached varied widely in soil-cement specimens containing soil from different depths in the Lloyd profile.

One relationship of the specimens is immediately obvious in Figure 3—less calcium hydroxide was leached from the specimens cured for 56 days than those cured for 2 days. This relationship indicates that calcium hydroxide was originially formed and
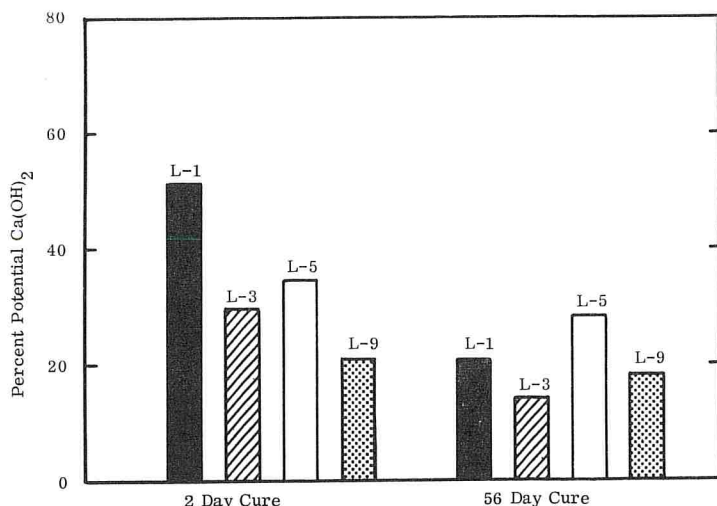

Figure 3. Percentage of potential Ca(OH)$_2$ leached from the Lloyd soil-cement specimens.

then was consumed in some manner. This consumption process was more pronounced in some specimens, especially the clay-rich ones, than in others (Table 7). The clay-size mineral suites of the Frederick and Lloyd specimens are so similar that they permit evaluation of the effect of kaolinite on the difference in quantity of leached calcium hydroxide with curing time (Table 7). Very small quantities of kaolinite were present in the Frederick specimens, and the difference in quantity of leached calcium hydroxide increased with curing time, i.e., calcium hydroxide accumulated. Kaolinite is the dominant clay in the Lloyd specimens, and the negative difference in quantity of calcium hydroxide leached became greater as the clay content increased. Thus the rate of calcium hydroxide consumption must have exceeded the rate of production, and did so to a greater extent with kaolinite than with any other clay mineral present.

If the typical B-horizon (L-1) and C-horizon (L-9) samples of the Lloyd soil are considered, 2 vastly different sets of calcium leachate data are available. (The Lloyd A-horizon, or leached horizon, has been eroded.) Approximately 52 percent of the potential calcium hydroxide was leached from soil-cement specimen L-1 after 2 days of curing, but only 21 percent of the potential was leached after 56 days of curing. From specimen L-9, however, only 21 percent of the potential was leached at 2 days and 18 percent at 56 days. This difference in the calcium leachate data indicates a difference in behavior of the B- and C-horizon soils when treated with portland cement. As pointed out earlier, neither sample appeared to hydrate at a normal rate, and therefore each has reacted with the cement in some manner. Large amounts of calcium hydroxide formed early in the B-horizon specimens indicating quick, almost accelerated, hydration of the cement. With further curing the calcium hydroxide appears to have been consumed and the calcium converted to a less leachable form, thus explaining the low 56-day leachate values. In the case of the C-horizon specimens, small amounts of the potential calcium hydroxide were leached after 2 days of curing and even smaller amounts at 56 days. This situation lends itself to either or both of 2 explanations: (a) Soil constituents may have prevented the initial formation of large quantities of calcium hydroxide; or (b) calcium hydroxide may have been produced in quantity but was rapidly consumed and the calcium tied up in less leachable componds.

The pH values of the Lloyd specimens varied widely, ranging from a low of 9.48 (specimen L-1, 56-day cure) to a high of 11.30 (specimen L-5, 56-day cure). Although a general trend prevailed, the pH did not always decrease or increase correlative to a decrease or increase in calcium leached from the 2-day to the 56-day cure. Soil properties must have affected the pH of the soil-cement systems in some manner; unfortunately, these properties and subsequently their effects could not be determined.

Leachings of Silica, Alumina, and Ferric Oxide Made With Hydrochloric Acid

The soil and soil-cement specimens were leached with 1.0 N HCl in order to determine the solubility of silica, alumina, and ferric oxide in the soils before and after cement treatment. It was thought that, if these componds were more or less acid soluble after cement treatment, some knowledge of the reactions occurring in the specimens might be derived. Whenever possible, information obtained from the water leachate and X-ray studies was correlated with the acid leachate results.

In the following discussion frequent reference is made to the calculated values given in Table 8. These values represent the amount of silica, alumina, or ferric oxide expected to be leached from each of the soil-cement specimens, assuming a normal rate of cement hydration and considering the dilution effect of the water of hydration. The values are the sum of the ppm of silica, alumina, or ferric oxide leached from the amount of soil in a soil-cement specimen plus the ppm of silica, alumina, or ferric oxide that it is theoretically possible to leach from the amount of cement in a soil-cement specimen, provided the cement is 50 percent hydrated. If normal type I portland cement hydration rates are considered, the specimens cured for 2 days would be approximately 50 percent hydrated, those cured for 21 days would be about 75 percent hydrated, and those cured for 56 days would be better than 80 percent hydrated (5). It is considered, therefore, that the assumption of 50 percent hydration of the cement in

TABLE 8

ACID LEACHINGS OF OXIDES FROM SOIL AND CALCULATED VALUES

Sample	Oxide	Soil Only (ppm)	Calculated Value[a] (ppm)	Sample	Oxide	Soil Only (ppm)	Calculated Value[a] (ppm)
F-1	SiO_2	5.4	193.4	L-1	Al_2O_3	9.9	61.9
F-2	SiO_2	3.2	191.2	L-3	Al_2O_3	7.2	59.2
F-4	SiO_2	4.8	192.8	L-5	Al_2O_3	7.2	59.2
F-8	SiO_2	3.5	191.5	L-9	Al_2O_3	8.8	60.8
F-12	SiO_2	1.6	189.6	F-1	Fe_2O_3	0.8	21.3
I-1	SiO_2	3.2	191.2	F-2	Fe_2O_3	4.8	25.3
I-2	SiO_2	7.2	195.2	F-4	Fe_2O_3	0.8	21.3
I-5	SiO_2	9.3	197.3	F-8	Fe_2O_3	4.8	25.3
L-1	SiO_2	6.4	194.4	F-12	Fe_2O_3	1.0	21.5
L-3	SiO_2	5.1	193.1	I-1	Fe_2O_3	2.4	24.9
L-5	SiO_2	4.8	192.8	I-2	Fe_2O_3	5.1	25.6
L-9	SiO_2	4.0	192.0	I-5	Fe_2O_3	2.9	23.4
F-1	Al_2O_3	8.2	60.2	L-1	Fe_2O_3	0.0	20.5
F-2	Al_2O_3	13.1	65.1	L-3	Fe_2O_3	1.3	21.8
F-4	Al_2O_3	13.6	65.6	L-5	Fe_2O_3	2.7	23.2
F-8	Al_2O_3	13.1	65.1	L-9	Fe_2O_3	2.1	22.6
F-12	Al_2O_3	10.4	62.4				
I-1	Al_2O_3	9.1	61.1				
I-2	Al_2O_3	13.9	65.9				
I-5	Al_2O_3	8.8	60.8				

[a] ppm possible from cement (50 percent hydrated) plus ppm leached from soil sample only.

the specimens is not unwarranted and for the last 2 curing periods is, in fact, a conservative estimate.

The data shown in Figures 4 through 12 are the ppm of silica, alumina, or iron oxide acid leached from the soil-cement specimens in excess of the calculated values.

It will be shown in the following discussions that all the soil-cement specimens leached more than the expected calculated values. This situation may be explained in 2 ways: (a) The cement in the specimen may not be 50 percent hydrated, thus resulting in greater than expected leachates of silica, alumina, and iron oxide because of the absence of predicted water of hydration; or (b) some of the clays in the soil may have slightly deteriorated releasing small quantities of silica, alumina, and iron oxide.

Frederick Soil-Cement Specimens—Acid leaches of the Frederick specimens again pointed up the uniformity of soil properties down the profile. Figures 4, 5, and 6 show that even at different curing periods all 5 soil-cement specimens leached almost the same amounts of silica, alumina, and ferric oxide.

Comparison of the leachate data of the Frederic specimens shows that in all instances the specimens leached practically the expected amounts of silica, alumina, and iron oxide. Because the leachate data were neither noticeably lower nor higher than the expected values, it must be concluded that the cement in the specimens hydrated at a normal rate and that there was little or no soil-cement reaction with its attendant deterioration of the clays.

However, 2 relationships of the lines may indicate some slight reaction of the soil with the cement: (a) Most of the curves show increased leachate values after 56 days of curing, thus indicating that limited deterioration of the soil components may have occurred with time; and (b) the specimens containing soil from the leached horizon (sample F-1) always leached less than the specimens made with soil from the zone of clay accumulation (sample F-2), thus implying that the clay zone in the Frederick is more susceptible to cement attack than the other horizons in the soil.

The reason for the very slight reaction of the soil constituents with the cement appears related to mineralogy. The major clay in the Frederick soil is illite. In previous studies concerned with this project (11), it was noted that of the relatively pure clays investigated the one showing least deterioration when mixed with cement was illite. It appears, therefore, that the apparent lack of soil-cement reactions in the Frederick specimens may be

related to the fact that the soil contains large amounts of the more stable clay, illite, instead of less stable minerals.

Iredell Soil-Cement Specimens—As was the case with the water leaches, the various horizons of the Iredell also yielded different acid leachate results. Figures 7, 8, and 9 show that when mixed with cement the leached horizon specimens (I-1) released much smaller quantities of silica, alumina, and iron oxide than did the B- and C-horizon specimens (I-2 and I-5).

The specimens made with soil from the Iredell leached zone (I-1), like all of the Frederick specimens, appear to have hydrated at a normal or almost normal rate with little soil-cement chemical interaction occurring. The amounts of silica, alumina, and iron oxide leached were only slightly higher than the expected calculated values. The apparent lack of chemical reactions appears related to weathering and the resultant mineralogy. The severe leaching of the Iredell A-horizon has developed a relatively stable mineralogical suite, with vermiculite, quartz, and illite

Figure 4. Excess silica acid leached from Frederick soil-cement specimens.

Figure 5. Excess alumina acid leached from Frederick soil-cement specimens.
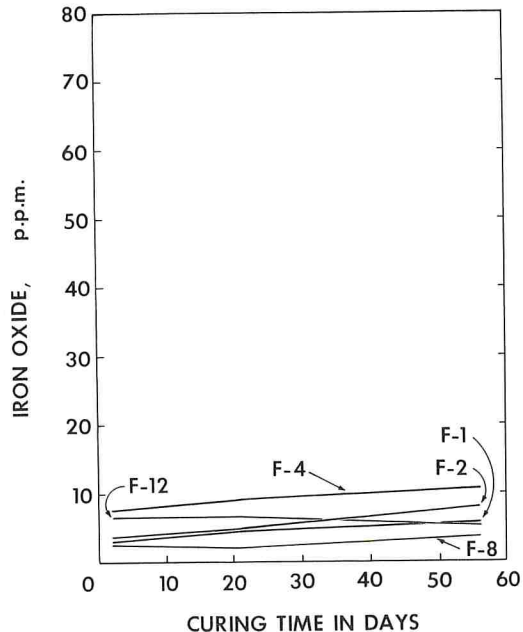
Figure 6. Excess iron oxide acid leached from Frederick soil-cement specimens.

respectively the principal clay-size minerals. If the Iredell A-horizon is in equilibrium with weathering conditions near the land surface, and if the cement environment is analogous to a weathering environment, albeit rigorous, then it follows that further breakdown and reaction of the soil with cement would not be expected.

The B-horizon soil-cement specimens (I-2), as shown in Figures 7, 8, and 9, leached much more silica, alumina, and iron oxide than the leached horizon specimens (I-1). When compared with the expected calculated values, the B-horizon soil-cement specimens leached an average of 89.1 ppm more silica, 28.5 ppm more alumina, and 22.9 ppm more iron oxide. Furthermore, the excess amounts leached increased with the length of the curing period. These excess amounts of silica, alumina, and iron oxide cannot be explained by slowed curing of the cement. In such a case lesser amounts of water of hydration would cause the calculated values to be higher, but not greatly higher. The excess amounts can be explained only by cement
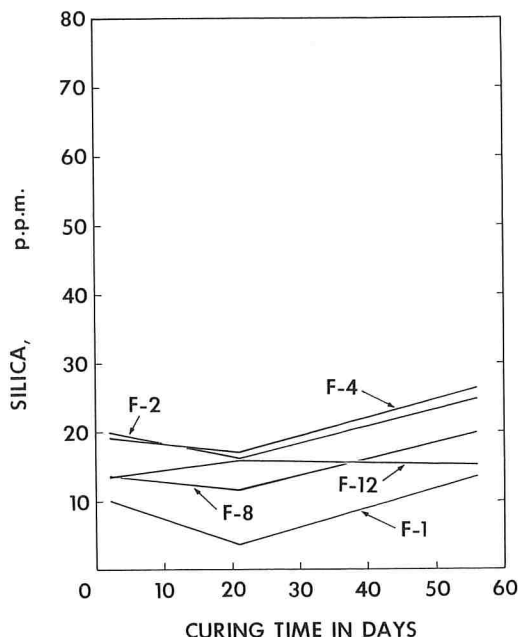

Figure 7. Excess silica acid leached from Iredell soil-cement specimens.

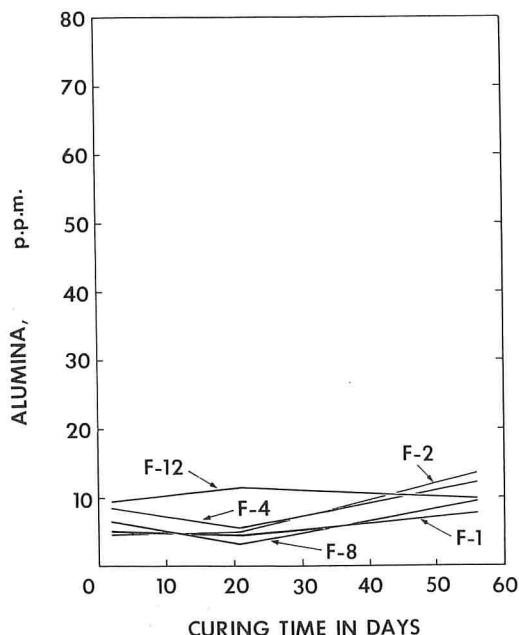

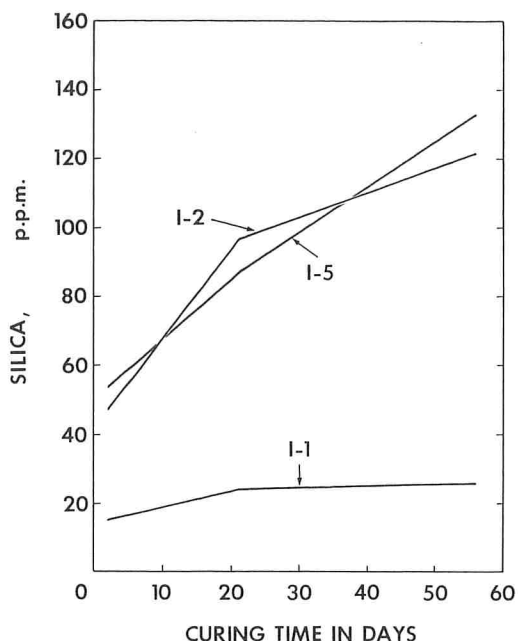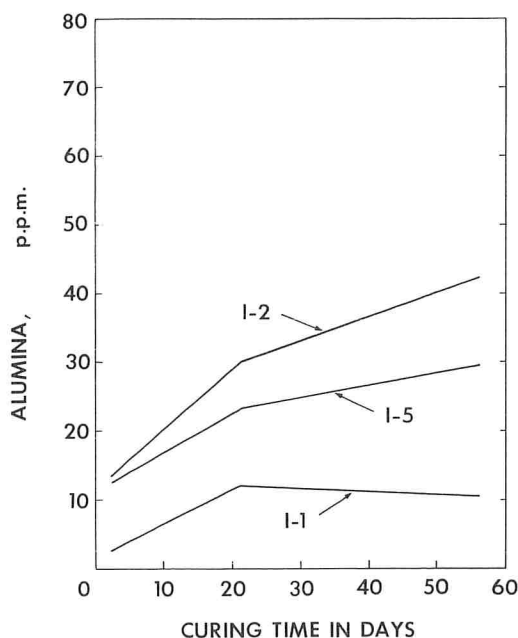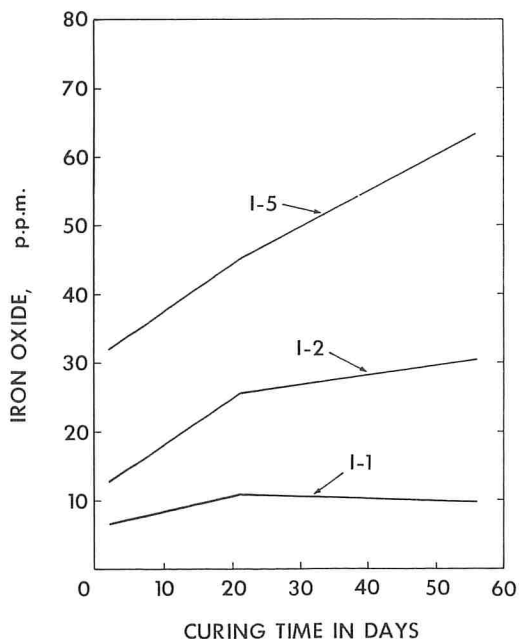Figure 8. Excess alumina acid leached from Iredell soil-cement specimens.

Figure 9. Excess iron oxide acid leached from Iredell soil-cement specimens.

attack and breakdown of the clays in the soil. The reactions possibly could have oc-
curred with amorphous materials, but the end result is the same—cement reaction has
caused these soil materials to deteriorate and become more acid leachable.

A relationship between the water leachate (calcium) data and the acid leachate (silica,
alumina, and iron oxide) data exists for specimen I-1 and other specimens described
later in which deterioration of the soil constituents appears to have occurred. The cal-
cium data were always low, and the silica and alumina data were always high; in some
instances the iron oxide data were also high. This relationship suggests that the clays
have deteriorated and that contemporaneous combination of the silica, alumina, and
iron oxide with calcium hydroxide has resulted in the formation of relatively insoluble
hydrated calcium silicates, aluminates, and ferrites.

The C-horizon soil-cement specimens (I-5) of the Iredell, like the B-horizon speci-
mens, yielded acid leachate values much larger than expected. Also, as in the B-horizon
specimens, the values increase with the curing time. Again it appears that soil-cement
reactions have caused silica, alumina, and iron in the soil to become more acid leach-
able. It would be interesting to postulate which soil constituents were involved. As was
noted earlier, although the C-horizon of the Iredell is a saprolite, the soil fraction used
in the leachate studies contained appreciable clay. This clay is largely montmorillonite,
which has been shown to deteriorate rapidly in a cement environment (9, 11). This de-
terioration would result in high silica and alumina (and possibly iron) concentrations
when the specimens were leached with acid. Also, in contrast to the leached zone of
the Iredell, the C-horizon has not undergone as severe weathering. The lower subsoil
is protected from downward seeping waters by an overlying impermeable clay zone.
The minerals in the C-horizon are not in equilibrium with surface conditions and would
be extremely susceptible to weathering. The harsh alkaline conditions of a cement en-
vironment might very easily cause deterioration of these minerals, again resulting in
high silica, alumina, and iron leachate values.

A final comment on the C-horizon specimens concerns the very high iron leachate
values, which increase with continued curing. Iron is abundant in the Iredell soil and
near the land surface has been oxidized into limonitic concretions. This oxidized iron
is not readily soluble, especially in an alkaline environment (7), and the low iron leach-
ate values of specimen I-1 would be expected. Lower in the soil profile, however, the
iron has not been oxidized and in the stagnant, poorly drained environment of the C-
horizon it probably is present in the more soluble ferrous state (8). It appears, there-
fore, that the ferrous iron originally dissolved in the specimens reacted with the ce-
ment and was precipitated as acid soluble compounds. More of the ferrous iron was
dissolved, reacted, and precipitated. This continuing process would explain the in-
creasingly high iron leachate values found for the C-horizon specimens. In order to
confirm this hypothesis, however, further work needs to be done on the hydration of
cement in a ferrous iron-rich solution.

Lloyd Soil-Cement Specimens—In the section on water leaches it was noted that the
different Lloyd specimens leached varied amounts of calcium. Figures 10, 11, and 12
show that the specimens also had varied acid leachate results. Also, it was stated that
the amount of calcium leached from the specimens by water decreased from the 2- to
the 56-day cure. With the exception of the silica data for specimen L-5, a reverse
trend is noted for the acid leachate results. The amount of silica, alumina, and iron
oxide leached increased with the curing time. Figures 10, 11, and 12 show that much
more than the expected amounts were leached from all of the specimens. This inverse
relationship between the water and acid leachate data seems to indicate, as was men-
tioned earlier, that calcium hydroxide formed in the specimens and then was consumed
in reactions with soil constituents. Thus, the amount of calcium leached would de-
crease with the usage of calcium hydroxide, and the amount of silica, alumina, and iron
oxide leached would increase with the formation of acid soluble reaction products.

If the specimens containing pure B-horizon soil (L-1) and those containing pure C-
horizon soil (L-9) are considered, it will be noted that they have similar leachate data.
The L-1 and L-9 curves shown in Figures 10, 11, and 12 roughly parallel each other,
illustrating this similarity. The large amounts of silica and alumina leached from both

sets of specimens after cement treatment indicate that the soil constituents have deteriorated in the cement environment. The very large amounts of alumina leached seem to indicate breakdown of the abundant kaolinite in the soil. In comparison with the Frederick and Iredell specimens, it appears that kaolinite is much more susceptible to cement attack than illite and practically as susceptible to cement attack as montmorillonite. This same ranking of pure clay minerals was found by Noble (11). The very high silica and alumina leachate data of specimen L-1 are easily explained because the soil used contained over 70 percent clay. The large amounts leached from the L-9 specimens, however, are more difficult to explain because the soil fraction used contained less than 15 percent clay. Deterioration of the clay may have contributed some of the excess silica and alumina, but breakdown of some of the primary minerals in the soil must also have taken place. Probably the easily weathered feldspar in the C-horizon was broken down by the cement.
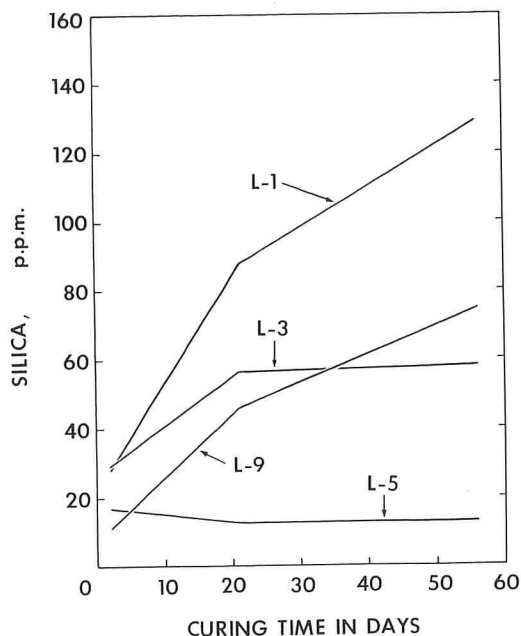

Figure 10. Excess silica acid leached from Lloyd soil-cement specimens.
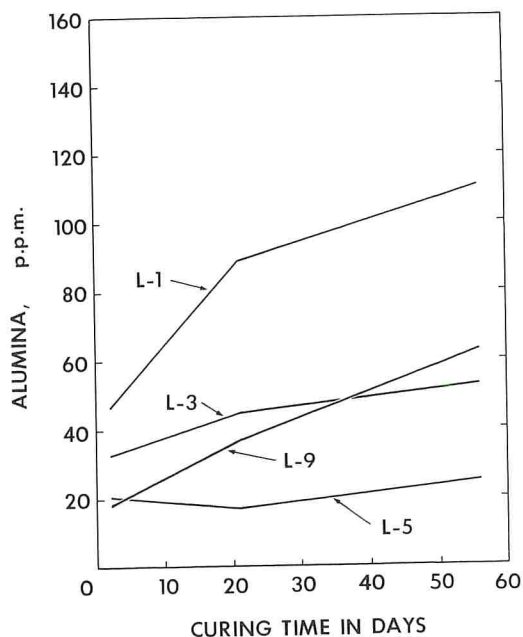

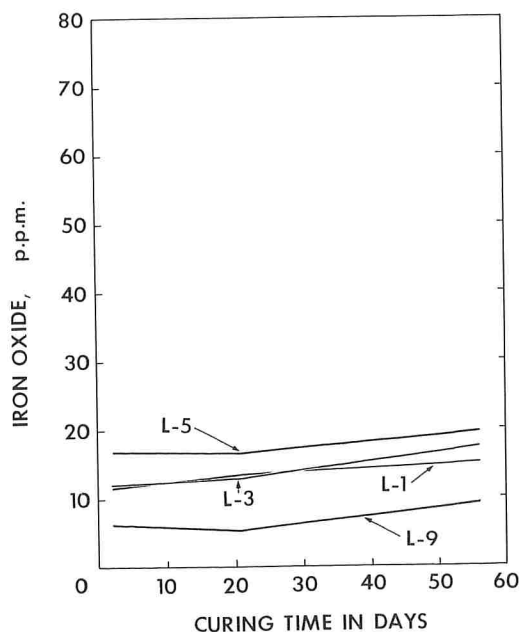Figure 11. Excess alumina acid leached from Lloyd soil-cement specimens.

Figure 12. Excess iron oxide acid leached from Lloyd soil-cement specimens.

X-Ray Investigations

 Unfortunately the slides prepared for this part of the study were X-rayed at different
times by different technicians using different diffractometer settings and possibly dif-
ferent diffractometer alignments. It was not possible, therefore, to immediately dis-
tinguish changes in relative X-ray intensities of the soil-cement constituents. Intensities
could be approximated, however, by using quartz occurring in the soils as an internal
standard; i.e., it was assumed that, in the presence of reactive clay minerals, the
quartz was nonreactive and did not change in X-ray intensity in any of the slides of the
same sample. In the following discourse, therefore, any reference to intensity changes
is based on the use of such a quartz standard.

 Also it should be pointed out that primary attention was focused on any changes in
crystallinity of the clays in the soils, because these minerals were thought the ones
most likely to react with cement.

 Frederick Soil-Cement Specimens—In the X-ray studies the uniformity of the Fred-
erick soil was again evident. There was little difference in the X-ray data of soil-
cement specimens prepared with soil from different profile depths. Consequently, all
of the Frederick specimens are treated collectively in this discussion.

 Illite, the major clay mineral of the soil, was present in both the 2- and 56-day
cured specimens, although its peak intensities were slightly diminished. Mixed layer
clay was evident in all of the specimens, but the basal d-spacings were more disordered
than in the untreated soil, indicating at least some lattice disruption. Kaolinite, the
third most abundant clay mineral in the soil, was noticeably diminished or absent in all
of the specimens. Basal kaolinite peaks were recognizable, but considerably reduced
in intensity in specimens F-2, F-8, and F-12 at 2 days, and were absent at 56 days.
The same peaks were absent at both 2 and 56 days in specimens F-1 and F-4.

 Although the masking effect of fine-grained cement hydration products may have af-
fected the X-ray reflections of the clay minerals, the data appear to indicate something
more. The slight peak intensity diminution of illite, the lattice disruption of mixed
layer clay, and the disappearance of kaolinite peaks are interpreted as evidence of ce-
ment attack of the clays. This evidence may account for the minor amount of soil de-
terioration that was postulated in the discussion of the leachate studies.

 Several cement hydration products were developed in the Frederick as well as the
Iredell and Lloyd soil-cement specimens. Identification of these compounds was ham-
pered, however, because of their poor crystallinity and the interference offered by
X-ray reflections of soil minerals. As a result, attention was focused on the better
crystallized compounds of calcium hydroxide and calcium monosulfoaluminate. Calcium
silicate hydrates of the tobermorite variety may have developed in all of the specimens,
but the definitive peaks of tobermorite were, in all cases, shadowed by the peaks of
other constituents.

 Calcium hydroxide was evident in all of the Frederick specimens after 2 days of cur-
ing and at 56 days showed even greater development. Because calcium hydroxide is a
major product of the hydration of cement, it seems that the presence of the Frederick
soil constituents has not greatly inhibited cement hydration. Instead it appears that hy-
dration has proceeded at a steady, if not increasing, rate. Calcium monosulfoaluminate
was developed in all of the specimens except F-1 at the 2-day cure. It was present,
however, in F-1 at 56 days and the 9 Å X-ray peak was increased in intensity at 56
days in specimens F-8 and F-12.

 Iredell Soil-Cement Specimens—Soil-cement specimens made with material from
different horizons of the Iredell soil had contrasting X-ray data.

 The most abundant clay in the leached horizon (specimen I-1) is vermiculite. After
2 days of curing this mineral persisted, but with reduced X-ray intensity. The same
situation prevailed at 56 days. The minor clay constituents of the soil, illite and chlo-
rite, were also slightly reduced in intensity at 2 and 56 days. The X-ray peaks of all
3 clays were more disordered after cement treatment than in the untreated samples,
indicating some deterioration of the clays. X-ray peaks of calcium hydroxide were
easily recognized and of about equal intensity in both the 2- and 56-day cured I-1 speci-
mens. Development of the compound appeared about the same as that in the Frederick

specimens, again demonstrating the similarity of reaction of the Iredell leached zone and the Frederick specimens. If calcium hydroxide production is again used as an index, the soil of the leached horizon showed more enhanced cement hydration than the 2 horizons lower in the Iredell profile.

The high clay content of the Iredell B-horizon showed a great effect on cement hydration. Also the X-ray diffractograms indicated that the clays had been substantially affected by the cement.

Illite, vermiculite, and chlorite were evident in the B-horizon specimens after 2 days of curing, but after 56 days only vermiculite was readily identified. Calcium hydroxide was not detectable at 2 days and appeared with only trace intensities at 56 days. These data might appear to indicate that only small amounts of calcium hydroxide formed. The diffractogram absence of calcium hydroxide, however, may not mean that it was never formed, but it may have been produced and then reacted with silica and alumina in the clay minerals. It is also possible that noncrystalline calcium hydroxide formed, which would not be detected by X-rays. Leachate data, however, will not support this latter hypothesis because only small amounts of calcium were leached from the specimens.

The C-horizon of the Iredell contained very little quartz. As a result, no standard was available for semiquantitative estimation of peak intensities. Nevertheless, it was obvious that montmorillonite, the major clay mineral of the C-horizon, was affected by the cement environment. The original 14.5 Å peak of the mineral appeared after cement treatment as a broad, disordered band of peaks ranging up to 17 Å. The increased d-spacing and resultant lattice expansion may indicate that cement attack was taking place between the mineral layers, or the expansion may simply have been due to the absorption of interlayer water. The low intensity illite peak at 10 Å originally present in the soil samples appeared unaffected after cement treatment, again illustrating the stability of illite in a cement environment.

Calcium hydroxide was developed in the C-horizon soil-cement specimens, but not nearly as well as in the leached zone specimens. It appeared about equally developed in both the 2- and 56-day cures. Calcium monosulfoaluminate was well developed in both the 2- and 56-day cures.

Lloyd Soil-Cement Specimens—X-ray data of the Lloyd specimens indicated that deterioration of some of the mineral constituents had occurred. Also, all 4 specimens showed some development of calcium hydroxide, but the compound appeared much less developed than in the Frederick specimens. The following is a discussion of the B-horizon (L-1) and C-horizon (L-9) soil-cement specimens.

In the B-horizon specimens kaolinite was considerably reduced in X-ray intensity, but the peaks were still easily discerned. The other major clay minerals in the B-horizon, mixed layer clay and illite, displayed very disordered X-ray peaks in diffractograms of the soil alone, and it was extremely difficult to determine if any changes had taken place after cement treatment. Calcium hydroxide displayed low intensity peaks after 2 days of curing and even lower ones after 56 days. The low peaks at 56 days are understandable because only small amounts of calcium were leached from specimen L-1 at that time. But the low peaks at 2 days were not expected because very large amounts of calcium were leached. It is possible that at 2 days large amounts of calcium hydroxide were present, but the compound had not crystallized and was largely in an amorphous state. It is also possible that the calcium hydroxide was entirely crystalline, but the abundant clays in specimen L-1 were absorbed onto the surface of the crystals. Thus, many of the X-ray reflections of the calcium hydroxide might be masked. Calcium monosulfoaluminate was slightly developed in the L-1 specimens at 2 days, but at 56 days it was absent.

X-ray diffractograms of the C-horizon specimens (L-9) were practically identical at the 2- and 56-day curing periods. Kaolinite peaks were greatly diminished in intensity, indicating deterioration of the clay. Illite was not reduced in intensity. Mixed layer clay was slightly diminished. Feldspar peaks were reduced in intensity, perhaps indicating that this mineral had reacted with the cement, as was postulated in the sections on leachates. Small amounts of calcium hydroxide were detected in the L-9 specimens, and calcium monosulfoaluminate was well developed.

Microscopic Studies

Petrographic investigations of thin sections showed the gross structure and general composition of the soil-cement specimens. Most of the constituents were extremely fine grained and, as a result, detailed studies of the soil-cement relationships could not be conducted.

In the Frederick sections some of the clay was flocculated, but generally clay, quartz, and cement products were diffusely distributed. Some of the quartz grains were coated with clay. A few patches of calcite were present and calcium hydroxide was developed.

Clay, quartz, biotite, calcium hydroxide, and a small amount of calcite were identified in the leached horizon section (I-1) of the Iredell specimens. The constituents were coarser grained than in the Frederick section, and the clay was highly flocculated. In several instances cementation of 2 mica shards by calcium hydroxide and other cement products had taken place. The cement products were often seen penetrating into the clay flocs.

The soil and cement constituents in the Iredell B-horizon section (I-2) were extremely fine grained. The clay was well flocculated into irregular masses, often including several quartz grains. In general, the cement surrounded the clay flocs, but in a few cases cement was present within the flocs. Calcium hydroxide was not identified.

Calcium hydroxide was not identified in the C-horizon specimens (I-5) of the Iredell. Hornblende, biotite, and chlorite were present. Very little of the clay was flocculated, but it was intimately mixed with the cement matrix. In one instance cement was noted eating into the edges of a biotite flake.

The clay was well flocculated in the B-horizon soil-cement section (L-1) of the Lloyd specimens. Illitic material appeared more abundant than X-ray studies had indicated. In some cases clay had coated quartz grains and surrounded patches of cement. The matrix was a mixture of clay, quartz, and cement. Calcium hydroxide was not identified.

Again calcium hydroxide was not identified in the C-horizon soil-cement section (L-9) of the Lloyd specimens. Very little clay was present in the slide, but quartz, feldspar, and mica were abundant. Many of the smaller soil particles were cemented together in isolated patches, and larger quartz and feldspar grains were often coated with cement.

Strength Development Studies

The results of the unconfined compressive strength tests are given in Table 9. The following observations were made:

1. Unconfined compressive strengths increased at least fourfold for the specimens after cement treatment, and in several instances increases were greater than twentyfold;

2. In general, the B-horizon specimens had lower strengths than the C-horizon specimens after cement treatment;

3. Specimen I-1, the only highly leached soil investigated, had a high strength after cement treatment;

4. Comparisons with the grain size distributions of the soils appear to indicate that the amount of clay in the soils was more important in determining short-term soil-cement strength gain than was the type of clay; and

5. No easily recognized relationship existed between the leachate and X-ray data of the soil-cement specimens and the development of strength. The strength gain of the specimens did not appear related to chemical reactions of the soil and cement.

TABLE 9

SEVEN-DAY UNCONFINED COMPRESSIVE STRENGTHS OF HARVARD-SIZE SOIL AND SOIL-CEMENT SPECIMENS

Sample	Strength (psi)	
	No Cement	10 Percent Cement
F-1	52.9	329.1
F-2	79.2	354.2
F-4	47.8	302.3
F-8	48.4	196.5
F-12	47.4	321.2
I-1	19.6	461.3
I-2	40.0	250.2
I-5	19.8	520.6
L-1	23.6	200.1
L-3	51.5	268.0
L-5	21.2	191.1
L-9	17.0	436.4

CONCLUSIONS

The following are some of the more important conclusions obtained from this study.

1. The Frederick soil constituents did not react appreciably with the cement, although some of the clays in the soil were slightly deteriorated after cement treatment.

2. As shown in the Frederick specimens, quartz neither impeded the production of nor contributed to the consumption of calcium hydroxide.

3. The clays and probably other constituents in the B- and C-horizons of the Iredell and Lloyd soils suffered deterioration after cement treatment, as indicated by the release of large amounts of silica and alumina and by the diminution of mineral X-ray peaks.

4. When the major clay mineral in the soils was considered, the soils appeared to suffer deterioration after cement treatment from greatest to least in the following order: montmorillonitic soils, kaolinitic soils, illitic soils.

5. The greatest quantities of calcium hydroxide were consumed with kaolinite as the dominant clay size mineral.

6. It appeared that reaction of calcium hydroxide with the soil constituents was proportional to the magnitude of the clay size fraction.

7. It appeared that amorphous calcium hydroxide was present in at least two of the specimens investigated.

8. The instability of the C-horizon Iredell soil constituents in a cement system appeared related to mineralogy. The minerals in the soil were not greatly weathered and were susceptible to the harsh conditions of an alkaline environment.

9. Iron in at least two of the Iredell specimens was highly reactive with or easily dissolved by the cement or both. This situation appeared related to the probable ferrous state of the iron.

10. The amount of clay in the soil-cement specimens appeared more important than the type of clay in determining short-term strength development.

ACKNOWLEDGMENTS

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Factors Affecting the Tensile Strength of Cement-Treated Materials

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Recent theoretical and analytical advances concerned with pavement failures due to excessive tensile stresses or strains or both within the soil-pavement system have placed added emphasis on the investigation of tensile properties of highway construction materials. This study evaluates the factors and interactions affecting the tensile characteristics of cement-treated materials. Nine factors were investigated: cement content, molding water content, aggregate gradation, curing time, curing temperature, type of aggregate, type of curing, type of compaction, and compactive effort. The first five were investigated at 3 levels and the last four at 2 levels in a statistically designed fractional factorial experiment. The parameter considered as a primary indicator of the tensile properties of cement-treated materials was the indirect tensile strength. An analysis of variance was used to determine the significance of all main factors, 2-factor interactions, and 3-factor interactions in the experiment. The highly significant effects ($\alpha = 0.005$) that are also of practical significance to the engineer are discussed in this paper, and tables of all additional factors and interactions significant at alpha levels of 1 and 5 percent are shown. A regression equation that predicts values of indirect tensile strength within the factor space defined by the experiment is presented.

•THE INTEREST in the tensile properties of highway construction materials is rapidly expanding. The development and refinement of layered system analyses (1, 2) have focused attention on the tensile and elastic properties of the materials used in the structural layers of a pavement system. A current failure hypothesis for highway pavements postulates that tensile cracking propagates from the bottom of a layer to the top. Analyses based on this hypothesis have verified its applicability but have been somewhat handicapped because information concerning the tensile properties of highway construction materials is practically nonexistent.

Historically, compressive testing techniques have been used to evaluate these materials, and as a result there is a great deal of information concerning their compressive strength but little information concerning their tensile strength. This paper summarizes one phase of an overall research effort to evaluate the tensile properties of stabilized subbase materials for use in the design of rigid pavements. The primary objectives of the investigation were to determine the factors that have an effect on the tensile strength of cement-treated materials, to determine the nature of the effect, and to develop a preliminary estimate of the tensile strengths that can be developed by treating a material with portland cement.

THE INDIRECT TENSILE TEST

The indirect tensile test was chosen as a means of evaluating the tensile characteristics of stabilized materials. Basically, this test involves the loading of a circular element with compressive loads acting along 2 opposite generators, as shown in Figure 1. This loading configuration produces a relatively uniform tensile stress distribution perpendicular to and along a portion of the diametral plane containing the applied load. Hudson and Kennedy (3) reported that, when the applied tensile stress exceeds the tensile strength of the stabilized materials, failure usually occurs by splitting along the load plane.

The equipment used in this study consisted of a closed loop electrohydraulic system and a loading head. The load frame was a modified, commercially available shoe die with upper and lower platens constrained to remain parallel during testing. The vertical deformation of the specimen was measured by a dc linear variable-differential transducer that also controlled the rate of load application. Test data were recorded in the form of a load-deformation relationship on an X-Y plotter. Additional information concerning the equipment and testing techniques can be found elsewhere (3, 4).
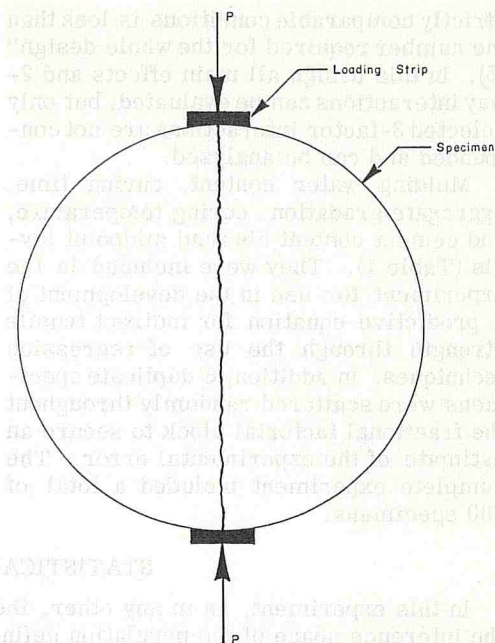

Figure 1. The indirect tensile test.

STATISTICAL DESIGN

Statistical methods were used extensively in the experimental design and analysis in order to obtain maximum information from a reasonable number of tests. Previous efforts concerned with evaluating the strength characteristics of cement-treated materials generally have limited the number of factors investigated at one time to two or three while attempting to hold constant any additional factors that were thought to affect the material. Such experiments, although providing a great deal of information on the factors investigated, do not allow the interaction effects of those factors being investigated with those held constant to be evaluated. (An interaction is the failure of 2 or more factors to act independently of each other; i. e., the rate of change of a variable with respect to one factor is dependent on the level of magnitude of one or more additional factors.) Thus it was felt that as many factors as possible should be included in a given experiment in order that interaction effects as well as main effects could be evaluated.

A detailed review of literature primarily involving compressive tests resulted in a list of factors that affect the strength of cement-treated materials. From this list, 9 factors considered to be important and of practical significance to the designer were selected. Each of these factors was included in the experiment at 2 or more representative levels (Table 1).

The use of full factorial experiment designs would have allowed all main effects and their interactions to be evaluated and analyzed. In this case, however, a complete factorial experiment could not easily be conducted because with 5 factors at 3 levels and 4 factors at 2 levels 3,888 specimens would have been required. Therefore, a one-fourth fractional factorial utilizing all factors at 2 levels was selected. By adopting this approach certain information was lost through confounding, a process "by which unimportant comparisons are deliberately confused for the purpose of assessing the more important comparisons with greater precision. Confounding is required in factorial experiments in which the number of observations capable of being carried out under

strictly comparable conditions is less than the number required for the whole design" (5). In this design all main effects and 2-way interactions can be evaluated, but only selected 3-factor interactions are not confounded and can be analyzed.

Molding water content, curing time, aggregate gradation, curing temperature, and cement content also had midpoint levels (Table 1). They were included in the experiment for use in the development of a predictive equation for indirect tensile strength through the use of regression techniques. In addition, 8 duplicate specimens were scattered randomly throughout the fractional factorial block to secure an estimate of the experimental error. The complete experiment included a total of 180 specimens.

TABLE 1

FACTORS AND LEVELS SELECTED

Factor	Level		
	Low	Medium	High
A. Molding water content, percent	3	5	7
B. Curing time, days	7	14	21
C. Aggregate gradation	Coarse	Medium	Fine
D. Type of curing	Air-dried	—	Sealed
E. Aggregate type	Gravel	—	Limestone
F. Curing temperature, deg F	40	75	110
G. Compactive effort	Low	—	High
H. Type of compaction	Impact	—	Gyratory shear
J. Cement content, percent	4	6	8

STATISTICAL INFERENCE

In this experiment, as in any other, the conclusions drawn are applicable only within the inference space of the population defined by the experiment design, i.e., within the range of combinations of values of the variables tested. No attempt should be made to apply the results outside of this particular inference space.

The principal objective of this study was to determine which factors significantly affect the tensile strength of cement-treated materials. Those factors or interactions found to significantly affect the tensile strength at probability levels of 0.005, 0.01, and 0.05 are given in Table 2. The effects of all other factors and interactions were not considered to have practical significance to the engineer. The residual shown in the table is the pooled mean squares for those factors and interactions that were not significant. The error mean square term was calculated from the duplicate specimens and represents an estimate of true error.

The relationships of the highly significant main factor interactions for tensile strength are shown in Figures 2 through 12. The data points in these figures are the average values of strength for all specimens containing a given level or combination of levels for the main effect or interaction. Midpoint means are not included in the figures or in the analysis of variance because the levels of the other factors are not the same as those for the high and low levels, and because the number of observations on the midpoint means cannot be compared to the endpoint means.

Because there were interactions that significantly affected the tensile strength, they must be considered in order to analyze the behavior associated with specific combinations of factors. It is not adequate to consider the main effects alone without evaluation of the interaction effects; in this paper interactions will be discussed first.

ANALYSIS OF VARIANCE

Data given in Table 2 show that 35 factors and their interactions were found to significantly affect the tensile strength of cement-treated materials at a probability level of 0.05 or greater, with 18 of these at a probability level of 0.005. Not all of these effects have practical significance; i.e., the effect may be measurable and under the controlled conditions of this test may be significant, but the effect is not large and probably makes little difference in the application of the results by engineers. Therefore, only those factors felt to be practically significant will be discussed.

Because 2- and 3-way interactions produced significant effects, it is mandatory that the higher order interaction effects be considered first, because any observed effect is the result of interrelationships among the various main factors. Thus, main effects can be referred to only in terms of the average effect because the effect is dependent

TABLE 2
ANALYSIS OF VARIANCE FOR TENSILE STRENGTH

Source of Variation[a]	Degree of Freedom	Mean Squares	F-Value	Significance Level (percent)
A	1	524,050	1480.00	0.5
D	1	142,607	404.00	0.5
J	1	127,391	361.00	0.5
E	1	108,056	306.00	0.5
AJ	1	57,196	162.00	0.5
AD	1	53,895	153.00	0.5
DE	1	34,340	97.30	0.5
G	1	29,248	82.90	0.5
EJ	1	23,795	67.40	0.5
AE	1	23,416	66.30	0.5
DJ	1	18,769	53.20	0.5
B	1	12,769	36.20	0.5
AC	1	11,012	31.20	0.5
H	1	8,430	23.90	0.5
ADJ	1	7,357	20.80	0.5
AH	1	6,992	19.80	0.5
EF	1	6,139	17.40	0.5
AB	1	5,798	16.40	0.5
DEJ	1	5,342	15.10	1.0
AFHJ-BCGJ	1	4,399	12.50	1.0
BG	1	4,337	12.30	1.0
EG	1	4,223	12.00	5.0
AEJ	1	4,212	11.90	5.0
BC	1	3,238	9.17	5.0
C	1	3,168	8.98	5.0
AEF	1	2,587	7.33	5.0
CDFJ	1	2,571	7.28	5.0
AEG	1	2,422	6.86	5.0
BDH	1	2,388	6.77	5.0
CEH	1	2,165	6.13	5.0
BEJ	1	2,128	6.03	5.0
BDFJ	1	2,077	5.88	5.0
BF	1	2,036	5.77	5.0
CH	1	2,013	5.70	5.0
BCJ	1	1,989	5.63	5.0
Residual	92	464		
Within treatments treated alike	7	353		

[a]A = molding water content   D = type of curing   G = compactive effort
B = curing time   E = aggregate type   H = type of compaction
C = aggregate gradation   F = curing temperature   J = cement content
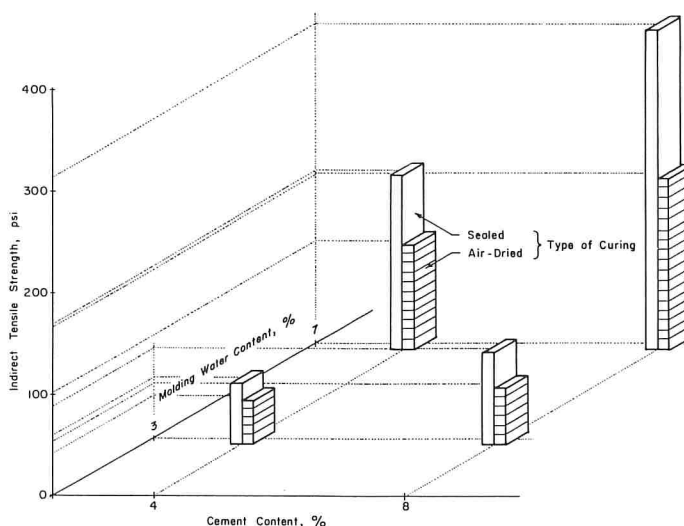

Figure 2. Effect of interaction among molding water content, type of curing, and cement content (interaction A X D X J).

on the interactions existing for any combination of factors. On this basis, significant higher order interactions will be discussed first and main effects last.

Three-Factor Interactions

A total of nine 3-factor interactions was found to be significant at a probability level of 0.05; however, only one was significant at a probability level of 0.005. This highly significant 3-factor interaction involved molding water content, type of curing, and cement content (interaction A x D x J).

As shown in Figure 2, the tensile strength increased from the point of low cement content and low molding water content to the point of high cement content and high molding moisture content; however, the increase was much greater for the increased moisture content than for increased cement content. Although the basic trends were the same regardless of the type of curing, the strength increases were much greater for specimens cured by sealing rather than air-drying.

Two-Factor Interactions

From a total of 36 analyzable 2-factor interactions, 15 were significant at a level of 0.05 or greater, with 10 of these significant at a level of 0.005. These 10 highly significant 2-way interactions are discussed below and are shown in Figures 3 through 12.

Molding Water Content and Cement Content (Interaction A x J)—It was observed that tensile strength increased with the increased molding water content (Fig. 3). The increase, however, was much greater for specimens containing the higher cement content. Likewise it can be noted that strength increased with increased cement content but that the increase was much greater for specimens compacted at a higher water content. Thus, it appears the beneficial effect of additional cement is limited unless there is an adequate supply of water for hydration of the cement.

Molding Water Content and Type of Curing (Interaction A x D)—The increase in the molding water content resulted in a greater increase in strength for the specimens that were sealed during the curing period than for the specimens cured by air-drying (Fig. 4). Such a phenomenon is logical because increased water would be expected to increase the efficiency of the hydration process. In the case of the sealed specimens this increased water was retained for hydration of the cement, but in the air-dried specimens it was lost; thus, its benefit was not fully realized.

Type of Curing and Aggregate Type (Interaction D x E)—It was found that the strength increase associated with changing the aggregate from gravel to limestone was much greater for the sealed specimens than for the air-dried specimens, although the strengths were greater for the limestone specimens in all cases (Fig. 5). As previously noted, when the specimens were cured by sealing, more water was available for hydration, and
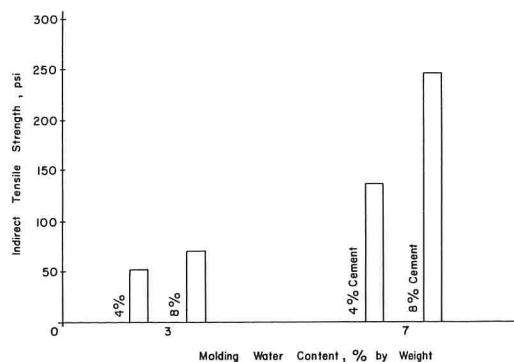

Figure 3. Effect of interaction between molding water content and cement content (interaction A X J).
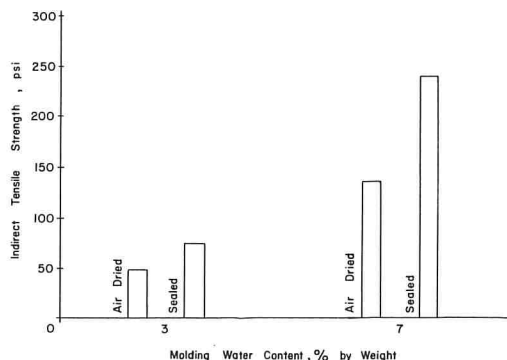
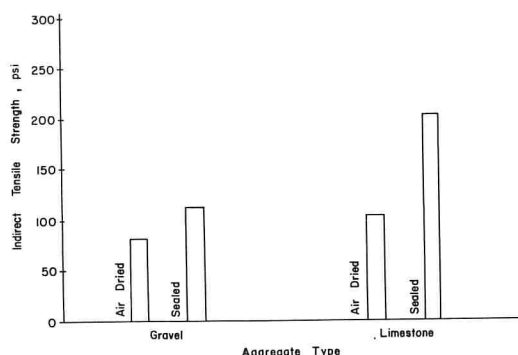Figure 4. Effect of interaction between molding water content and type of curing (interaction A X D).

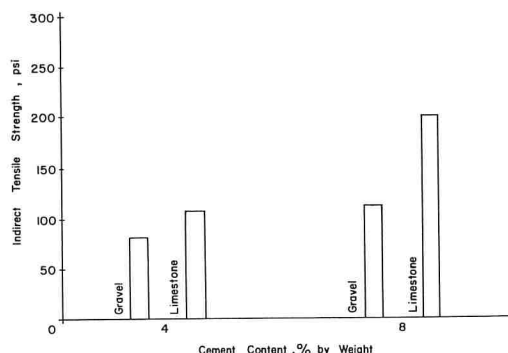Figure 5. Effect of interaction between type of curing and aggregate type (interaction D X E).

Figure 6. Effect of interaction between aggregate type and cement content (interaction E X J).

the result was an improved cement matrix. Apparently, the benefits of the improved matrix are more fully realized by the limestone aggregate, which can develop a better cement-aggregate bond due to its angularity and rough surface texture.

Aggregate Type and Cement Content (Interaction E x J)—The strength increase associated with the increased cement content was greater for specimens containing limestone than for those containing gravel (Fig. 6). It is felt that this interaction effect illustrates once again that the limestone is able to benefit more from a stronger matrix than the rounded gravel.

Molding Water Content and Aggregate Type (Interaction A x E)—The strength increase associated with the increase in water content was greater for the limestone than for the gravel (Fig. 7). As in the case of the interaction of type of curing and aggregate type (Fig. 5), the limestone benefited more than the gravel from the improved matrix resulting from better hydration.

Type of Curing and Cement Content (Interaction D x J)—The average strength increased with the increase in cement content in all specimens whether cured by sealing or by air-drying (Fig. 8). The strength increase, however, was much greater for the sealed specimens than for the air-dried specimens. Thus, the benefit of the increased cement content was more fully realized when the specimens were cured under the more ideal curing conditions.
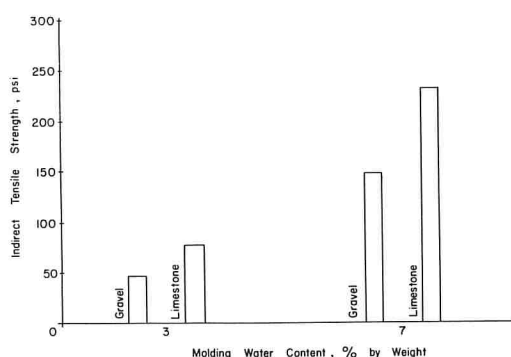

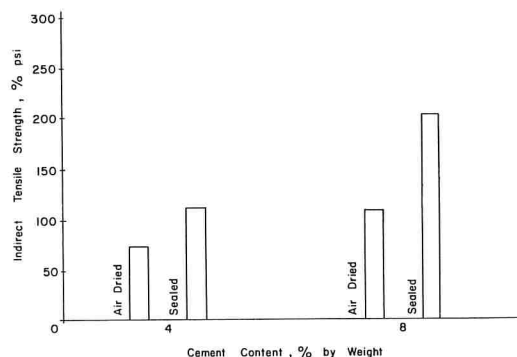Figure 7. Effect of interaction between molding water content and aggregate type (interaction A X E).

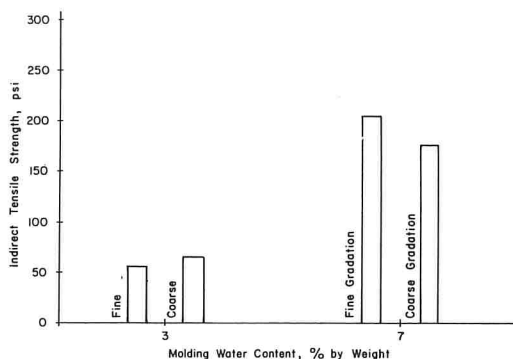Figure 8. Effect of interaction between type of curing and cement content (interaction D X J).

Figure 9. Effect of interaction between molding water content and aggregate gradation (interaction A x C).

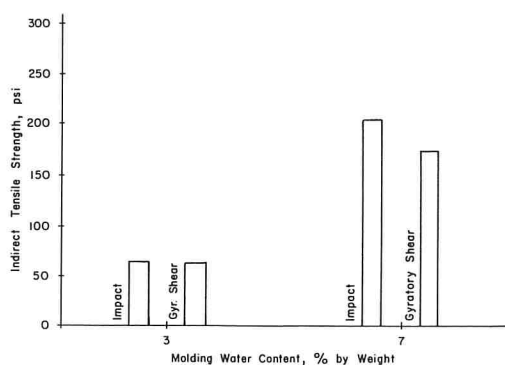Figure 10. Effect of interaction between molding water content and type of compaction (interaction A x H).

Molding Water Content and Aggregate Gradation (Interaction A x C)—It was found that a molding water content of 7 percent produced stronger specimens than 3 percent water but that the increase in strength was much greater for specimens containing a finely graded aggregate (Fig. 9). In addition, a change from finely graded aggregate to a coarse-graded aggregate produced a strength increase for specimens compacted at 3 percent water, but the reverse was true for specimens compacted at 7 percent water.

Molding Water Content and Type of Compaction (Interaction A x H)—As in the previous interaction, the increased molding moisture content resulted in higher strengths; however, the amount of this increase was dependent on the type of compaction, with impact compacted specimens producing a greater increase in strength than the gyratory shear specimens.

Curing Temperature and Aggregate Type (Interaction E x F)—It appears that the increased curing temperature, from 40 to 110 F, produced higher tensile strengths for specimens containing gravel; however, there was little effect on the strength of the limestone specimens (Fig. 11).

Molding Water Content and Curing Time (Interaction A x B)—Increased water content at the time of molding resulted in stronger specimens. Although specimens cured for 21 days seemed to have larger strength gain than specimens cured 7 days, Figure 12
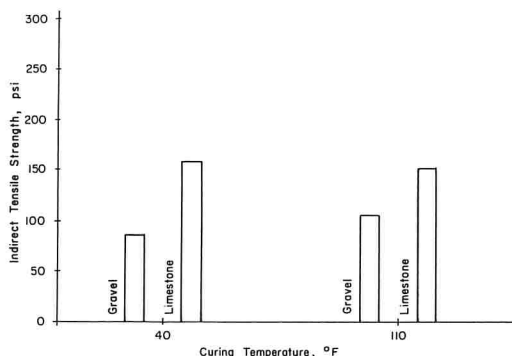

Figure 11. Effect of interaction between aggregate type and curing temperature (interaction E X F).
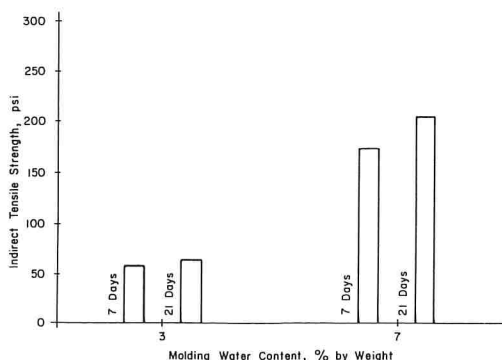

Figure 12. Effect of interaction between molding water content and curing time (interaction A X B).

shows that the difference in the rate of increase associated with the 2 different curing times is very small and of little practical significance.

Main Effects

Seven of the 9 main effects were found to be significant at a probability level of 0.005. It was found that the average indirect tensile strength was significantly increased by (a) increasing the molding water content from 3 to 7 percent (factor A); (b) sealed rather than air-dried curing (factor D); (c) increasing the cement content from 4 to 8 percent (factor J); (d) using crushed limestone rather than rounded gravel aggregates (factor E); (e) using a high compactive effort (factor G); (f) curing for 21 days rather than 7 days (factor B); and (g) using impact compaction rather than gyratory shear compaction (factor H).

Evaluation and Discussion

This experiment was designed to investigate, but not necessarily to explain, the causes of the effects produced by all 9 factors and their interactions. Nevertheless, it is desirable and possible to postulate the causes of the observed behavior and, in almost all cases, to advance logical explanations for future consideration. By comparing the relative values of the mean squares (Table 2), it can be seen that the water content during mixing and compaction was by far the most important factor affecting strength. In addition to its highly significant main effect, it is also involved in 6 highly significant 2-factor interaction effects and one 3-factor interaction effect. It would appear that this effect is primarily concerned with the hydration process rather than with compaction or mixing, because in 4 of the 6 highly significant 2-way interactions, molding water content is associated with a factor concerned with hydration, i.e., cement content, type of curing, aggregate type, and curing time. In these interactions it can be reasoned that the interaction is the result of improved hydration or the ability of the soil to benefit from improved hydration. Thus, the strength gain for increased molding water content was greater for higher cement contents, was greater for sealed specimens in which water was retained for hydration, was greater for limestone than for gravel because the rough surface texture of the limestone could develop a better bond with the improved matrix, and was greater for the longer curing time, which allowed better hydration to occur. The other 2 highly significant 2-way interactions involved aggregate gradation and type of compaction. Both had relatively low mean squares and were considered to be relatively unimportant in comparison to most other highly significant effects involving molding water content.

It may be noted that of the 9 factors chosen for investigation because of their apparent importance, 7 were found to produce highly significant effects. The remaining 2 factors, aggregate gradation and curing temperature, were judged to produce no effects of practical significance although aggregate gradation indicated some significance at the probability level of 0.05. It is not surprising that curing temperature did not produce a significant main effect because there was evidence that water needed for hydration was removed from the specimens at the upper level of temperature (110 F): Droplets of moisture condensed on the inside surface of the PVC wrap used to seal the specimens. Thus, the beneficial effect of increased curing temperature was offset by the loss of water associated with the increase in temperature. In general, any factor that could be expected to increase the strength of the cement matrix or improve the bond between the cement matrix and soil particles resulted in increased strengths.

REGRESSION ANALYSIS

A stepwise regression analysis was conducted in order to develop a predictive equation for indirect tensile strength. The high and low levels as well as the applicable intermediate levels of all factors were used as input data. From this analysis a predictive equation was developed allowing the indirect tensile strength to be estimated within some standard error for the inference space defined by this experiment. The regression equation obtained for the indirect tensile strength is

$$S_T = -110.85 - 21.35E_i + 20.68A_iJ_i + 1.25A_iE_iJ_i + 11.31D_iE_i$$
$$- 4.70A_i^2 - 1.63A_iJ_i^2 + 0.15A_i^2D_i + 0.30A_i^2H_i + 1.22A_iC_i$$
$$- 0.05B_i^2G_i$$

where

S_T = predicted value of indirect tensile strength, in psi,

A, B, C, D, E, G, H, J = factors considered for prediction, and

i = level of the factor (Table 3).

The multiple correlation coefficient for the tensile strength predictive equation is $R = 0.95$, and the standard error of the estimate is equal to ± 32.0. The lack of fit of this regression equation is not significant at a probability alpha level of 0.05 and is not considered critical. The equation is only valid for predictive purposes within the factor space studied, which is a function of all factors and levels studied. Any attempt to extrapolate beyond the factor space with the regression equation may of course introduce conditions not investigated here and may result in erroneous predictions.

Furthermore, the factors and interactions included in the predictive equation are not identical to those shown to be highly significant by the analysis of variance. This is partially because of the fact that an additional level for each factor was included in the regression analysis; thus the data for the 2 analyses were not the same. A second cause is that the predictive equation is concerned only with those variables and combinations of variables that provide the best estimate of the dependent variable. Hence, if 2 independent variables (factors) are highly correlated, the regression analysis may include only one of them.

TABLE 3

LEVELS OF FACTORS USED IN
REGRESSION EQUATIONS

Factor	Description	Level
A. Molding water content	3 percent	$A_0 = 3$
	5 percent	$A_1 = 5$
	7 percent	$A_2 = 7$
B. Curing time	7 days	$B_0 = 7$
	14 days	$B_1 = 14$
	21 days	$B_2 = 21$
C. Aggregate gradation	Coarse	$C_0 = 0$
	Medium	$C_1 = 1$
	Fine	$C_2 = 2$
D. Type of curing	Air-dried	$D_0 = 0$
	Sealed	$D_2 = 2$
E. Type of aggregate	Rounded gravel	$E_0 = 0$
	Crushed limestone	$E_2 = 2$
F. Curing temperature	40 F	$F_0 = 40$
	75 F	$F_1 = 75$
	110 F	$F_2 = 110$
G. Compactive effort	Low	$G_0 = 0$
	High	$G_2 = 2$
H. Type of compaction	Rainhart impact	$H_0 = 2$
	Gyratory shear	$H_2 = 0$
J. Cement content	4 percent	$J_0 = 4$
	6 percent	$J_1 = 6$
	8 percent	$J_2 = 8$

SUMMARY

The results of this study of tensile properties of cement-treated materials can be summarized as follows:

1. Seven of the 9 factors evaluated produced significant main effects on the indirect tensile strength at a probability level of 0.005.

2. Ten of the 36 two-factor interactions evaluated produced significant effects on the indirect tensile strength at a probability level of 0.005.

3. One 3-factor interaction was found to be significant at a probability level of 0.005.

4. The large number of interactions significant at a probability level of 0.005 indicate the complexity of the relationships between tensile strength and the factors involved.

5. In addition to these highly significant effects, 1 main effect, 4 two-factor interactions, 7 three-factor interaction effects, and 2 four-factor interaction effects were found to be significant at a level of 0.05.

6. Curing temperature was the only factor that did not produce a significant main effect at a level of 0.05. This should not be interpreted to mean that curing temperature was not important because there was evidence that moisture needed for hydration was driven from the specimens cured at 110 F, and the benefits of increased temperature may have been offset by the loss of moisture.

7. Molding water content was the most important factor affecting the strength of the

cement-treated materials because it was a highly significant main effect and was involved in 6 of the 10 highly significant 2-factor interaction effects.

8. Increased molding water content resulted in significantly higher strengths apparently because of improved hydration rather than improved compaction.

9. In general, any factor that could be expected to increase the strength of the cement matrix or improve the bond between the cement matrix and soil particles resulted in increased strengths.

10. In evaluating the effects produced by various factors, it is not adequate to infer only from main effects; rather one must consider the interactions between the factors involved, in order to predict tensile strength.

ACKNOWLEDGMENTS

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Appendix 1

TYPES OF GRADATIONS USED IN THE EXPERIMENT

COARSE GRADATION - Type A

Sieve Size	Texas Highway Department Specifications,* Percent by Weight	Gradation Used, Percent by Weight
Passing 2 inch	100	100
Passing 1-3/4 inch	95-100	100
Passing 1-3/4 inch Retained 7/8 inch	15-40	0

Passing 7/8 inch Retained 3/8 inch	15-40	26
Passing 3/8 inch Retained No. 4	10-25	21
Passing No. 4 Retained No. 10	5-20	15
Total Retained on No. 10	65-80	62
Passing No. 10 Retained No. 40	0-20	15
Passing No. 40 Retained No. 80	3-15	5
Passing No. 80 Retained No. 200	2-15	8
Passing No. 200	0-8	10
Passing 1/2 inch	100	98
Passing 3/8 inch	95-100	95
Passing 3/8 inch Retained No. 4	20-50	20
Passing No. 4 Retained No. 10	10-30	15
Total Retained No. 10	60-75	40
Passing No. 10 Retained No. 40	0-30	30
Passing No. 40 Retained No. 80	4-25	10
Passing No. 80 Retained No. 200	3-25	10
Passing No. 200	0-8	10

*"Standard Specifications for Road and Bridge Construction," Texas Highway Department, 1962.

Appendix 2

TREATMENT COMBINATIONS

Specimen Number	Test Order	Level of Factor[1]								
		A	B	C	D	E	F	G	H	J
1	91	-1	+1	-1	+1	-1	-1	-1	+1	+1
2	36	0	0	-1	-1	-1	0	-1	-1	0
3	1	-1	-1	-1	-1	-1	-1	-1	-1	-1
4	92	+1	+1	-1	-1	-1	-1	-1	-1	+1
5	93	-1	+1	-1	-1	+1	-1	-1	+1	-1
6	94	0	+1	0	+1	+1	0	+1	+1	0
7	2	-1	-1	-1	+1	+1	+1	-1	+1	+1
8	37	0	0	0	-1	-1	0	-1	-1	+1
9	3	+1	-1	-1	-1	+1	+1	+1	+1	+1
10	95	+1	+1	-1	+1	+1	-1	+1	+1	-1
11	96	+1	+1	+1	-1	+1	-1	-1	+1	+1
12	97	+1	+1	+1	+1	-1	-1	-1	+1	-1
13	38	0	0	0	-1	-1	0	-1	-1	0
14	98	-1	+1	-1	+1	-1	+1	-1	-1	+1
15	99	-1	+1	+1	+1	+1	-1	-1	-1	-1
16	4	+1	-1	+1	-1	-1	-1	+1	+1	-1
17	39	0	0	+1	+1	+1	0	+1	+1	0
18	40	0	0	0	+1	+1	0	+1	+1	0
19	5	-1	-1	-1	+1	+1	-1	-1	-1	-1
20	100	-1	+1	-1	-1	+1	+1	+1	+1	+1
21	6	+1	-1	-1	-1	+1	-1	-1	+1	-1
22	101	+1	+1	+1	+1	-1	-1	+1	-1	+1
23	102	+1	+1	-1	+1	+1	+1	+1	-1	-1
24	103	-1	+1	+1	+1	+1	-1	+1	+1	+1
25	104	+1	+1	-1	+1	+1	+1	-1	+1	+1
26	7	0	-1	0	+1	+1	0	+1	+1	0
27	105	-1	+1	+1	+1	+1	+1	+1	-1	+1
28	8	+1	-1	+1	-1	-1	+1	-1	+1	+1
29	106	+1	+1	-1	+1	+1	-1	-1	-1	+1
30*	41	0	0	0	-1	-1	0	-1	-1	0
31	9	+1	-1	+1	-1	-1	-1	-1	-1	+1
32	107	-1	+1	+1	+1	+1	+1	-1	+1	-1
33	108	0	+1	0	-1	-1	0	-1	-1	0
34	109	-1	+1	-1	+1	-1	+1	+1	+1	-1
35	10	+1	-1	+1	+1	+1	-1	-1	-1	-1
36	11	-1	-1	-1	+1	+1	-1	+1	+1	-1
37	12	-1	-1	+1	-1	+1	+1	+1	+1	-1
38	13	-1	-1	+1	-1	+1	-1	-1	+1	+1
39	14	+1	-1	+1	+1	+1	-1	+1	+1	+1

40	15	-1	-1	+1	-1	+1	+1	-1	-1	+1
41	110	-1	+1	-1	-1	+1	-1	+1	-1	+1
42	16	-1	-1	+1	+1	-1	-1	-1	+1	-1
43	42	+1	0	0	+1	+1	0	+1	+1	0
44	43	-1	0	0	-1	-1	0	-1	-1	0
45	17	-1	-1	+1	+1	-1	+1	-1	-1	-1
46	18	0	-1	0	-1	-1	0	-1	-1	0
47	44	0	0	0	-1	-1	+1	-1	-1	0
48	19	-1	-1	+1	+1	-1	-1	+1	-1	+1
49	20	+1	-1	+1	-1	-1	+1	+1	-1	-1
50	45	0	0	0	+1	+1	0	+1	+1	+1
51	46	0	0	0	+1	+1	+1	+1	+1	0
52	47	0	0	-1	+1	+1	0	+1	+1	0
53	111	-1	+1	-1	-1	+1	+1	-1	-1	-1
54	112	+1	+1	+1	+1	-1	+1	+1	+1	+1
55	113	+1	+1	+1	-1	+1	+1	+1	+1	-1
56	21	+1	-1	+1	+1	+1	+1	+1	-1	-1
57	114	+1	+1	-1	-1	-1	+1	-1	+1	-1
58	48	0	0	0	-1	-1	0	-1	-1	-1
59	49	0	0	0	+1	+1	0	+1	+1	-1
60	22	+1	-1	-1	-1	+1	+1	-1	-1	-1
61	23	-1	-1	+1	+1	-1	+1	+1	+1	+1
62*	50	0	0	0	+1	+1	0	+1	+1	0
63	51	0	0	0	-1	-1	-1	-1	-1	0
64	115	+1	+1	+1	+1	-1	+1	-1	-1	-1
65	24	-1	-1	-1	-1	-1	+1	-1	+1	-1
66	116	+1	+1	+1	-1	+1	+1	-1	-1	+1
67	52	0	0	+1	-1	-1	0	-1	-1	0
68	25	-1	-1	+1	-1	+1	-1	+1	-1	-1
69	26	-1	-1	-1	+1	+1	+1	+1	-1	-1
70	117	-1	+1	+1	-1	-1	+1	-1	+1	+1
71*	118	+1	+1	+1	-1	+1	+1	-1	-1	+1
72	119	+1	+1	-1	-1	-1	-1	+1	+1	+1
73	27	+1	-1	-1	+1	-1	+1	-1	-1	+1
74	120	-1	+1	+1	-1	-1	+1	+1	-1	+1
75	28	+1	-1	-1	+1	-1	+1	+1	+1	-1
76	29	+1	-1	+1	+1	+1	+1	-1	+1	-1
77	30	-1	-1	-1	-1	-1	+1	+1	-1	+1
78	53	0	0	0	+1	+1	-1	+1	+1	0
79	121	-1	+1	+1	-1	-1	-1	-1	-1	+1
80*	31	+1	-1	-1	+1	-1	+1	+1	+1	-1
81	54	-1	0	0	+1	+1	0	+1	+1	0
82	32	+1	-1	-1	+1	-1	-1	-1	+1	+1
83	33	+1	-1	-1	-1	+1	-1	+1	-1	+1

84	122	-1	+1	-1	+1	-1	-1	+1	-1	-1
85	123	+1	+1	+1	-1	+1	-1	+1	-1	-1
86	34	+1	-1	-1	+1	-1	-1	+1	-1	-1
87	35	-1	-1	-1	-1	-1	-1	+1	+1	+1
88	55	+1	0	0	-1	-1	0	-1	-1	0
89	124	+1	+1	-1	-1	-1	+1	+1	-1	+1
90	125	-1	+1	+1	-1	-1	-1	+1	+1	-1
91	126	+1	0	0	+1	+1	0	+1	+1	0
92	56	+1	-1	+1	+1	+1	-1	+1	+1	-1
93	145	+1	+1	+1	+1	-1	-1	+1	-1	-1
94	57	-1	-1	+1	-1	+1	+1	-1	-1	-1
95	58	-1	-1	+1	-1	+1	-1	+1	-1	+1
96	146	-1	+1	-1	-1	+1	+1	+1	+1	-1
97	147	+1	+1	-1	+1	+1	+1	-1	+1	-1
98	148	+1	+1	+1	-1	+1	+1	-1	-1	-1
99	59	+1	-1	+1	+1	+1	+1	+1	-1	+1
100	60	+1	-1	+1	-1	-1	+1	-1	+1	-1
101	61	-1	-1	+1	+1	-1	-1	+1	-1	-1
102	149	+1	+1	-1	+1	+1	-1	+1	+1	+1
103	150	-1	+1	+1	-1	-1	+1	+1	-1	-1
104	151	-1	+1	+1	+1	+1	-1	+1	+1	-1
105	127	0	0	0	+1	+1	+1	+1	+1	0
106	62	-1	-1	+1	+1	-1	+1	+1	+1	-1
107	152	-1	+1	+1	-1	-1	-1	-1	-1	-1
108	63	+1	-1	+1	+1	+1	-1	-1	-1	+1
109	64	0	-1	0	+1	+1	0	+1	+1	0
110	153	-1	+1	+1	+1	+1	-1	-1	-1	+1
111	128	0	0	0	+1	+1	-1	+1	+1	0
112	154	-1	+1	+1	-1	-1	-1	+1	+1	+1
113	65	-1	-1	-1	+1	+1	-1	+1	+1	+1
114	66	+1	-1	+1	-1	-1	-1	-1	-1	-1
115	129	0	0	0	-1	-1	0	-1	-1	-1
116	67	+1	-1	-1	+1	-1	-1	-1	+1	-1
117	130	0	0	0	-1	-1	-1	-1	-1	0
118	68	+1	-1	-1	+1	-1	-1	+1	-1	+1
119	155	-1	+1	-1	-1	+1	-1	+1	-1	-1
120	69	-1	-1	+1	-1	+1	-1	-1	+1	-1
121	70	+1	-1	-1	-1	+1	+1	+1	+1	-1
122	156	0	+1	0	-1	-1	0	-1	-1	0
123	131	0	0	0	-1	-1	0	-1	-1	0
124	71	-1	-1	+1	-1	+1	+1	+1	+1	+1
125	157	-1	+1	-1	+1	-1	-1	-1	+1	-1
126	158	+1	+1	-1	-1	-1	+1	-1	+1	+1
127	159	+1	+1	+1	-1	+1	+1	+1	+1	+1

128	160	+1	+1	+1	+1	-1	+1	-1	-1	+1
129	132	+1	0	0	-1	-1	0	-1	-1	0
130	161	+1	+1	-1	+1	+1	+1	+1	-1	+1
131	72	-1	-1	-1	-1	-1	+1	-1	+1	+1
132	162	+1	+1	-1	-1	-1	-1	+1	+1	-1
133	73	-1	-1	-1	-1	-1	+1	+1	-1	-1
134	163	+1	+1	+1	-1	+1	-1	-1	+1	-1
135	74	+1	-1	-1	+1	-1	+1	-1	-1	-1
136	75	-1	-1	-1	+1	+1	-1	-1	-1	+1
137	76	0	-1	0	-1	-1	0	-1	-1	0
138	133	0	0	-1	-1	-1	0	-1	-1	0
139	134	0	0	0	-1	-1	+1	-1	-1	0
140	77	+1	-1	-1	-1	+1	-1	+1	-1	-1
141	78	-1	-1	+1	+1	-1	-1	-1	+1	+1
142	164	-1	+1	+1	+1	+1	+1	-1	+1	+1
143	165	-1	+1	+1	+1	+1	+1	+1	-1	-1
144	166	+1	+1	+1	+1	-1	+1	+1	+1	-1
145*	79	-1	-1	-1	+1	+1	-1	+1	+1	+1
146	80	+1	-1	-1	-1	+1	-1	-1	+1	+1
147	81	-1	-1	+1	+1	-1	+1	-1	-1	+1
148	167	+1	+1	+1	+1	-1	-1	-1	+1	+1
149	135	0	0	0	-1	-1	0	-1	-1	+1
150*	168	-1	+1	+1	+1	+1	-1	+1	+1	-1
151	82	+1	-1	+1	-1	-1	+1	+1	-1	+1
152	169	0	+1	0	+1	+1	0	+1	+1	0
153	83	+1	-1	-1	-1	+1	+1	-1	-1	+1
154	84	+1	-1	+1	+1	+1	+1	-1	+1	+1
155*	170	0	+1	0	-1	-1	0	-1	-1	0
156	171	+1	+1	-1	+1	+1	-1	-1	-1	-1
157	136	0	0	+1	-1	-1	0	-1	-1	0
158	172	-1	+1	+1	-1	-1	+1	-1	+1	-1
159	173	-1	+1	-1	-1	+1	+1	-1	-1	+1
160	174	-1	+1	-1	+1	-1	+1	+1	+1	+1
161	137	0	0	-1	+1	+1	0	+1	+1	0
162	175	-1	+1	-1	+1	-1	+1	-1	-1	-1
163	176	+1	+1	-1	-1	-1	-1	-1	-1	-1
164	85	-1	-1	-1	-1	-1	-1	-1	-1	+1
165	138	0	0	+1	+1	+1	0	+1	+1	0
166	86	+1	-1	-1	+1	-1	+1	+1	+1	+1
167	177	+1	+1	+1	-1	+1	-1	+1	-1	+1
168	139	-1	0	0	+1	+1	0	+1	+1	0
169	178	-1	+1	-1	-1	+1	-1	-1	+1	+1
170	87	-1	-1	-1	+1	+1	+1	-1	+1	-1
171	140	-1	0	0	-1	-1	0	-1	-1	0

172	88	-1	-1	-1	+1	+1	+1	+1	-1	+1
173	141	0	0	0	+1	+1	0	+1	+1	-1
174	179	-1	+1	-1	+1	-1	-1	+1	-1	+1
175	180	+1	+1	-1	-1	-1	+1	+1	-1	-1
176*	142	0	0	0	-1	-1	0	-1	-1	-1
177	143	0	0	0	+1	+1	0	+1	+1	0
178	144	0	0	0	+1	+1	0	+1	+1	+1
179	89	+1	-1	+1	-1	-1	-1	+1	+1	+1
180	90	-1	-1	-1	-1	-1	-1	+1	+1	-1

*Duplicate specimens.

[1]Level of Factor

-1 Low Level
 0 Middle Level
+1 High Level

Appendix 3

EXPERIMENTAL RESULTS

Specimen No.	Indirect Tensile Strength, psi	Specimen No.	Indirect Tensile Strength, psi
1	34.7	46	74.3
2	126.4	47	113.7
3	20.3	48	39.4
4	174.6	49	72.2
5	14.3	50	225.4
6	248.9	51	272.5
7	123.3	52	243.3
8	140.5	53	25.3
9	212.0	54	238.0
10	257.2	55	91.6
11	174.6	56	242.6
12	90.5	57	103.1
13*	139.0	58	111.3
14	39.3	59	187.7
15	54.1	60	101.9
16	50.0	61	84.9
17	268.9	62*	206.5
18*	252.0	63	105.7
19	57.8	64	183.5
20	66.6	65	40.4
21	103.8	66*	190.5
22	237.9	67	137.6
23	290.7	68	60.4
24	197.5	69	99.6
25	366.5	70	32.3
26	260.2	71*	202.6
27	157.5	72	134.0
28	115.6	73	283.2
29	431.8	74	70.9
30*	129.3	75*	127.7

31	118.0	76	114.8
32	80.8	77	70.5
33	89.8	78	212.9
34	44.0	79	37.0
35	169.6	80*	145.4
36	74.2	81	102.1
37	41.6	82	139.8
38	68.0	83	221.7
39	364.1	84	57.0
40	57.7	85	162.4
41	86.4	86	131.1
42	27.0	87	45.2
43	316.0	88	169.4
44	41.1	89	251.8
45	53.7	90	100.1
91	375.2	136	102.8
92	196.8	137	122.6
93	117.1	138	113.4
94	47.1	139	106.9
95	41.4	140	147.2
96	44.8	141	48.0
97	175.8	142	77.0
98	96.0	143	132.9
99	385.3	144	159.1
100	63.7	145*	82.3
101	26.0	146	147.5
102	497.1	147	55.0
103	54.3	148	207.4
104*	70.9	149	113.9
105	259.2	150*	74.5
106	71.8	151	122.0
107	41.1	152	170.3
108	374.3	153	128.2
109	254.1	154	365.1
110	106.3	155*	93.5
111	245.6	156	197.8
112	54.7	157	104.5
113*	105.6	158	26.4
114	63.8	159	59.5
115*	98.1	160	74.7
116	108.5	161	192.3
117	98.0	162	37.0
118	283.3	163	123.0
119	62.0	164	13.1
120	40.6	165	303.8
121	93.1	166	247.5
122*	136.8	167	291.2
123	115.5	168	108.3
124	63.7	169	73.1
125	30.9	170	40.4
126	127.1	171	42.0
127	227.6	172	146.4
128	142.4	173	233.5
129	157.8	174	43.6
130	495.5	175	162.9
131	39.6	176*	92.4
132	88.0	177	280.2
133	50.3	178	252.8
134	125.9	179	68.0
135	180.3	180	22.4

*Duplicate specimens.

Use of Sodium Chloride in Reducing Shrinkage in Montmorillonitic Soil-Cement

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Shrinkage in soil-cement results in progressive cracking and has been the cause of increasing concern. This paper reports the results of an experimental investigation to reduce shrinkage in Na-montmorillonitic soil-cement by using sodium chloride as a trace additive. The mechanism of shrinkage derived from this study was extended to Ca-montmorillonite and was also found generally applicable. Shrinkage was found to decrease greatly with increasing salt content except when used as a form of brine; in one case shrinkage was eliminated completely and actual expansion was observed. Strength reductions in the soil-cement specimens with sodium chloride additions were found to be due only to the increasing coarseness of the additive and not to the amount used. Addition of 1 to 3 percent fine-grained salt was found to produce strength comparable to that of soil-cement. The reduction of shrinkage in soil-cement with addition of salt was found to be primarily due to the ability of salt to reduce moisture loss in the mixture and to provide a more favorable and stable clay basal spacing. Particle reorientation during curing was also believed to be responsible for some of the shrinkage and expansion observed.

•SHRINKAGE IN SOIL-CEMENT is known to result in progressive cracking and has been the cause of increasing concern. This cracking is due to the tensile and shear stresses that are developed when shrinkage is partially or fully restrained. These cracks could conceivably become the cause of poor long-term engineering performance, if left untreated.

Examination of the factors that influence the behavior of shrinkage in soil-cement yields the following (3, 6):

1. Shrinkage results primarily from loss of water due to evaporation. The rate of evaporation decreases with time mainly because of increasing bonding energy of the remaining water with decreasing moisture content and decreasing size of water-filled capillaries. Nevertheless, evaporation of held water causes much greater shrinkage per gram evaporated, because it results in increased internal tensions in the remaining water.

2. Shrinkage of soil-cement is a function of the cement content and, when plotted, it exhibits a minimum at an optimum cement content. Increasing shrinkage of soil-cement with increasing cement content above optimum was presumed to be due to the greater water requirement of the cement to complete hydration. Thus cement hydration results in desiccation and shrinkage, and this shrinkage increases with cement content.

3. The clay content has also been known to increase shrinkage in soil-cement. This is due to the fineness of the -2 micron particles that have a large quantity of bonded water and hence result in large shrinkage as the water evaporates. It is also possible

that the clay content constitutes a matrix that is restrained less by the proportionally fewer +2 micron particles that act as rigid inclusions.

4. The kind of clay present in the soil-cement influences the amount of shrinkage; montmorillonite contributes the most for being the finest of all clay minerals. In addition, evaporation of the interlayer water in montmorillonite results in decreasing lattice spacing of the clay mineral. This type of shrinkage is naturally much greater than the shrinkage that results simply from evaporation of pore water.

5. Shrinkage increases with molding moisture wet of the Proctor optimum moisture content. The reason was explained to be that higher moisture contents make the -2 micron particles more apt to change from a cardhouse, flocculated structure to an oriented, dispersed one. When evaporation occurs, the latter structure allows for more shrinkage because of its weakly restrained particles.

The purpose of this study was to investigate the feasibility of reducing shrinkage in montmorillonitic soil-cement based on the concepts of lattice expansion of montmorillonites and retention of moisture with the addition of sodium chloride. Hopefully, it will also help in further delineating the shrinkage mechanism in montmorillonitic soil-cement.

METHODS AND PROCEDURES

Materials

The clay used was a sodium montmorillonite, commercially produced as Volclay by the American Colloid Company. Its exchangeable sodium and calcium cations range from 60 to 65 me/100 g and 5 to 10 me/100 g respectively ([1]). Ottawa sand, type I portland cement, and distilled water were the other constituents used together with the Volclay to prepare soil-cement specimens.

Commercially available rock salt for de-icing purposes with more than 99 percent NaCl was used. Different percentages were used in molding soil-cement specimens. The commercial gradation and the gradations of the salt used in the laboratory are given in Table 1.

Preparation and Curing of Specimens

The composition of the control specimens was as follows:

Material	Percent by Weight
Ottawa sand	52
Volclay	32
Portland cement	16

The mixing moisture content was 23.5 percent, based on the total dry weight of solids in the mix. This composition was found to give best workability throughout the process.

Different percentages of salt, based on the dry weight of the control mix, were added to mold soil-cement specimens in order to study the effect of the additive.

Soil-cement specimens of various proportions were molded in batches of 4 specimens. All batches were dry-mixed first for 1 minute to ensure uniform distribution of all constituents. The batches were then wet-mixed for 2 minutes. All mixing was done in a Hobart mixer, Model C-100.

The test specimens were prepared by weighing a constant amount of mix and placing it into a mold. The mold consists of a stainless steel tube (I.D. = 2.00 in.) and 2 end-plungers. The mix was statically compacted into a cylindrical specimen

TABLE 1

GRADATIONS OF SALT

Sieve	Percent Passing			
	Commercial	Coarse	Medium	Fine
⅜ in.	100.0			
¼ in.	89.2	100.0		
No. 4	58.8			
No. 6	24.2			
No. 8	6.7			
No. 10	2.8	0.0	100.0	
No. 40			0.0	100.0

2 in. in diameter and 4 in. in height. This procedure provides a constant dry density of 105 lb/cu ft for all specimens.

After the extrusion of the specimen, a small plate of galvanized sheet metal was attached at one end. At the center of the other end, a small triangular metal chip was attached. These attachments were made to increase accuracy and reproducibility of measurements and were made rigid by using a minimal amount of quick-setting glue.

All specimens were cured at 90 percent relative humidity, ±5 percent, and 25 C, ±1 deg. The curing took place in the cabinet of an incubator.

Measuring and Testing

Changes in specimen height with time were measured by using a dial gage with sensitivity of 0.0001 in. This gage was mounted on a rigid frame that provides a smooth, horizontal base for seating of the specimen. Accurate measurement of the specimen height was taken at the center of its metal chip.

At the end of the specified curing period, all specimens were tested for unconfined compression strength in order to study possible strength reductions caused by the added salt.

X-Ray Diffraction

Determination of changes in lattice spacing was done using a General Electric XRD-5 diffractometer with CuK α radiation. Powder samples were pressed into brass rings using static compaction of 1,000 psi. This helps to obtain dense packing and to eliminate preferred orientation. The ring was also rotated in the X-ray beam to increase reproducibility (7).

RESULTS

Effects of Salt on Shrinkage

Shrinkage versus time graphs are shown in Figures 1 through 7. Each point on the graphs represents the average of readings from 4 specimens cured up to 7 days.
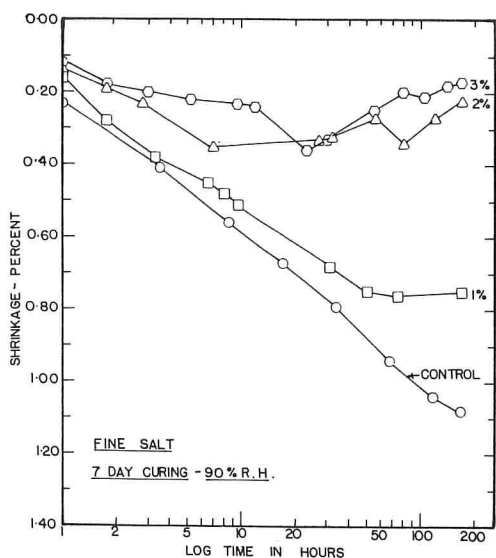

Figure 1. Shrinkage versus time for Na-montmorillonitic soil-cement with various percentages of fine salt and cured under 90 percent relative humidity for 7 days.
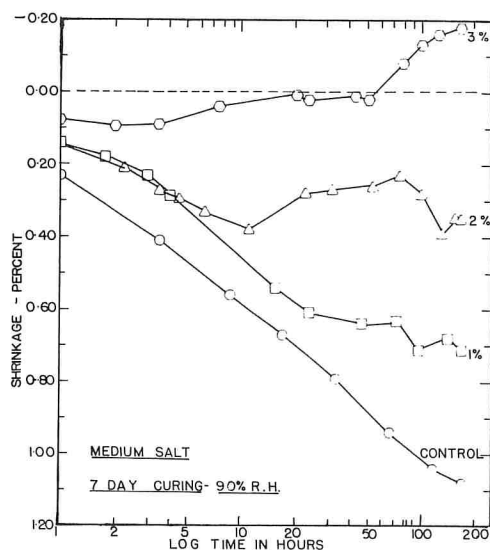

Figure 2. Shrinkage versus time for Na-montmorillonitic soil-cement with various percentages of medium salt and cured under 90 percent relative humidity for 7 days.
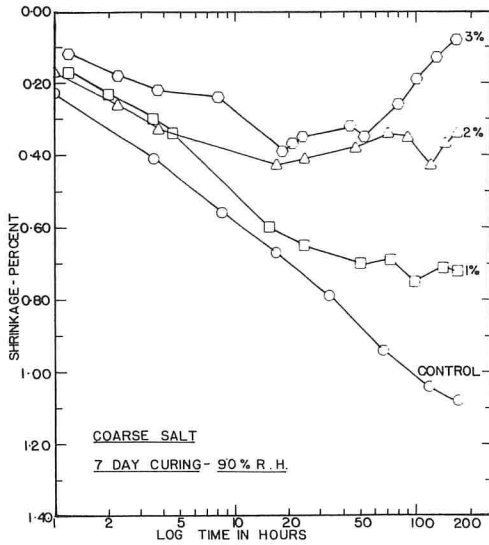
Figure 3. Shrinkage versus time for Na-montmorillonitic soil-cement with various percentages of coarse salt and cured under 90 percent relative humidity for 7 days.
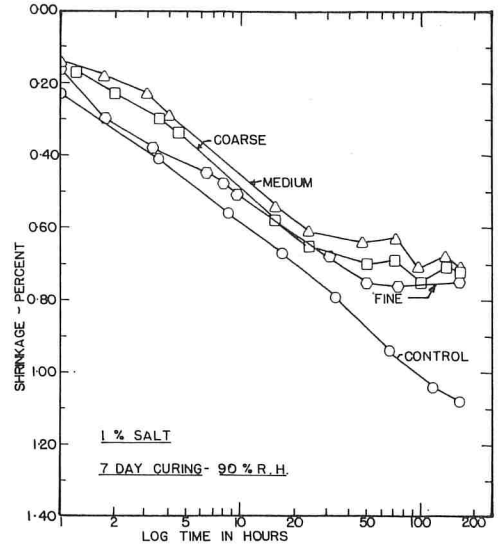

Figure 4. Shrinkage versus time for Na-montmorillonitic soil-cement with 1 percent salt of various gradations and cured under 90 percent relative humidity for 7 days.

Figure 5. Shrinkage versus time for Na-montmorillonitic soil-cement with 2 percent salt of various gradations and cured under 90 percent relative humidity for 7 days.

Figure 6. Shrinkage versus time for Na-montmorillonitic soil-cement with 3 percent salt of various gradations and cured under 90 percent relative humidity for 7 days.
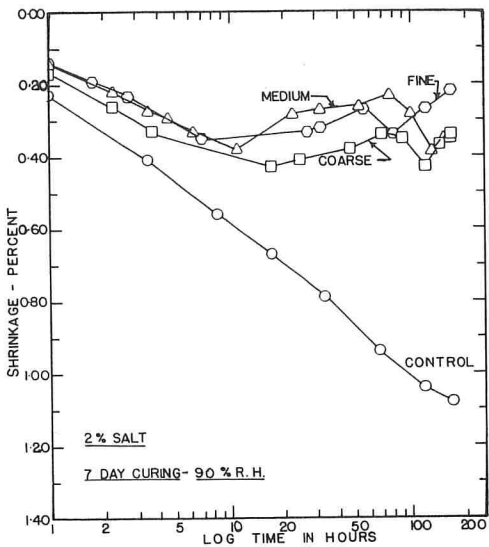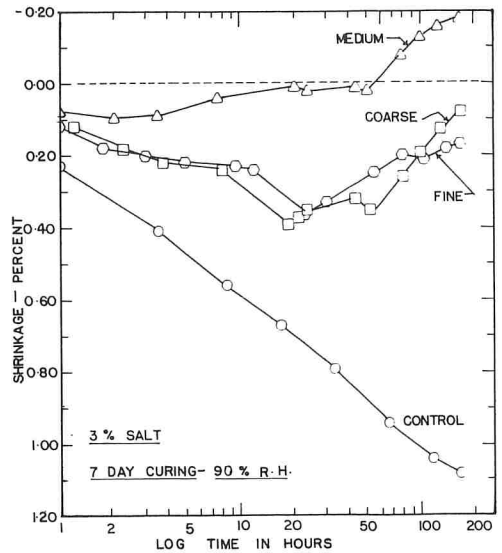
Figures 1, 2, and 3 show that shrinkage decreases greatly with increasing salt content of any of the gradations used. Net expansion was observed when 3 percent medium salt was added to the soil-cement specimens. However, different gradations of the same percentages of salt added did not produce a good enough trend of results to allow any conclusions as to their relative effect, as shown in Figures 4, 5, and 6.

Addition of 0.5 percent salt in solution form to the standard specimens did not reduce shrinkage appreciably, as shown in Figure 7.

An analysis of variance study was performed to determine the statistical significance to the effect of salt on shrinkage (2). Bands of 95 percent confidence uncertainty limits were established around the means of 4 specimens of the various batches throughout the curing time. Significant difference in amounts of shrinkage between any 2 batches is indicated by cessation of overlapping and subsequent separation of their corresponding bands. Figure 8 shows this comparison in which 10 hours is the curing time that amounts

Figure 7. Shrinkage versus time for Na-montmorilonitic soil-cement with 0.5 percent salt added in solution form and cured under 90 percent relative humidity for 7 days.

of shrinkage between the soil-cement control specimens and the soil-cement specimens with 2 percent coarse salt become significantly different. Figure 9 shows calculated times of significant differences in shrinkage between control specimens and specimens
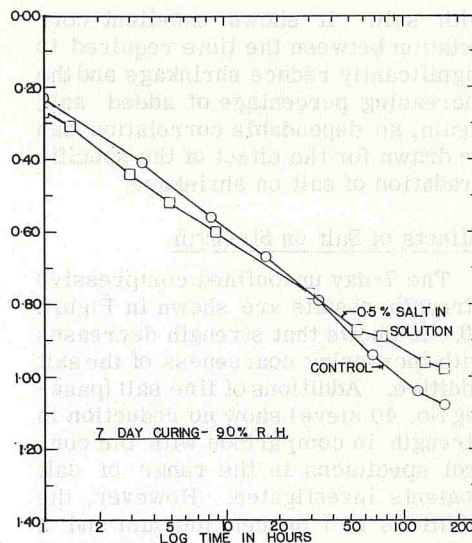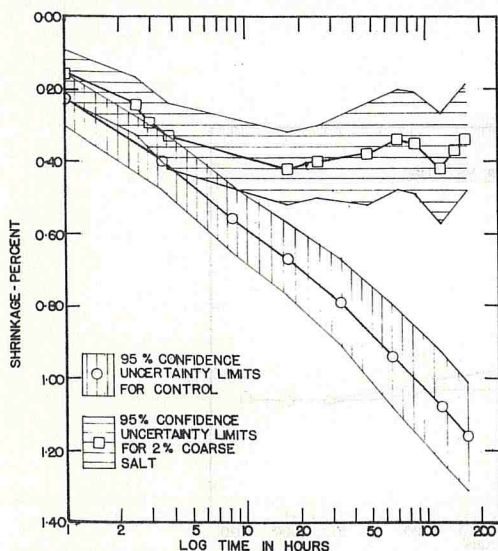

Figure 8. Comparison of 95 percent confidence uncertainty limits for shrinkage versus time.
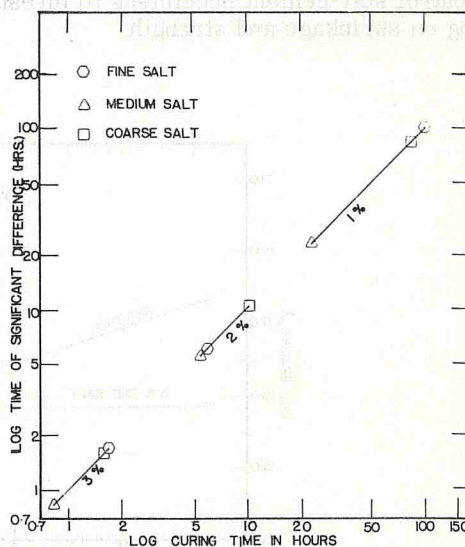

Figure 9. Time of significant difference in shrinkage between soil-cement control specimens and specimens with salt.

with salt. It shows excellent correlation between the time required to significantly reduce shrinkage and the increasing percentage of added salt. Again, no dependable correlation can be drawn for the effect of the specific gradation of salt on shrinkage.

Effects of Salt on Strength

The 7-day unconfined compressive strength results are shown in Figure 10. It shows that strength decreases with increasing coarseness of the salt additive. Additions of fine salt (passing No. 40 sieve) show no reduction in strength in comparison with the control specimens in the range of salt contents investigated. However, the additions of 3 percent medium and 3 percent coarse salts resulted in 14 and 34 percent strength reductions respectively.

Figure 10. Comparison of 7-day unconfined compressive strength of soil-cement control specimens with specimens containing salt.

X-Ray Investigation

Figure 11 shows that while the basal (001) spacing of the montmorillonite in soil-cement specimens with salt remained practically constant, the 001 spacing of the clay in the control specimens decreased steadily throughout the curing period. Similar corresponding behavior was also observed in the variation of moisture content in the specimens throughout the curing period, as shown in Figure 12.

Effects of Immersion and 28-Day Curing

Soil-cement samples containing 3 percent medium salt were also compared with the control soil-cement specimens to investigate the effects of immersion and 28-day curing on shrinkage and strength.
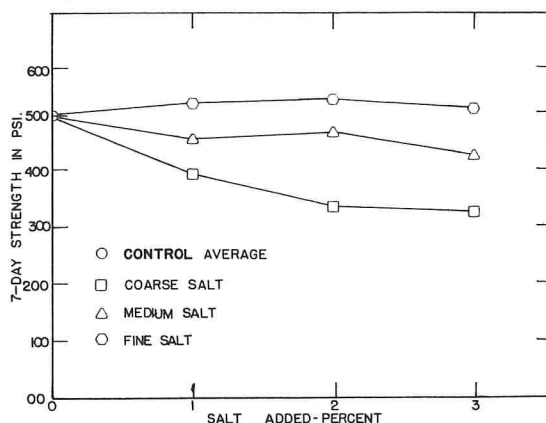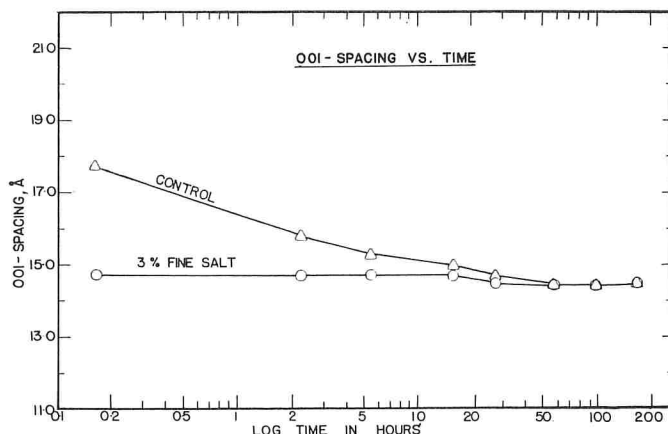

Figure 11. Basal (001) lattice spacing of Na-montmorillonite versus curing time in soil-cement specimens.
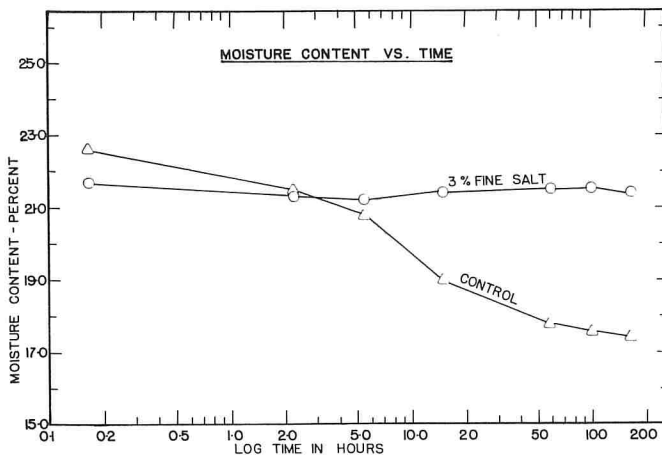
Figure 12. Moisture content versus curing time in soil-cement specimens.

Results have shown that samples with salt continually maintained a lower rate of shrinkage than the control specimens under regular curing at 90 percent relative humidity and 25 C up to 28 days (dotted lines in Fig. 13). However, upon immersion after 7 days of regular curing, the control soil-cement specimens swelled significantly, while the samples with salt were practically unaffected, as shown with the solid lines in Figure 13.

Results also showed that 28-day strengths followed the same order as the 7-day strengths for the regularly cured specimens. However, specimens that were regularly cured for 7 days and then immersed for the rest of the 28-day curing period showed comparable strengths for the soil-cement specimens with and without salt.

Figure 13. Shrinkage versus time for Na-montmorillonitic soil-cement with 3 percent medium salt and cured under various conditions for 28 days.

Results From Using Ca-Montmorillonite

All the results presented so far have been obtained from specimens molded with Na-montmorillonite. To check the effectiveness of using sodium chloride also as a shrinkage reducing agent in Ca-montmorillonite soil-cement, 0 and 3 percent medium salt specimens similar to those used previousy were molded using calcium instead of sodium montmorillonite. The Ca-clay used is known as the Panther Creek Southern Bentonite, commercially produced by the American Colloid Company.

Shrinkage and strength results similar to those shown in Figures 2 and 10 were also obtained. Furthermore, soil-cement specimens containing salt again maintained a practically constant moisture content throughout the curing period, while specimens without salt showed a drying tendency similar to that
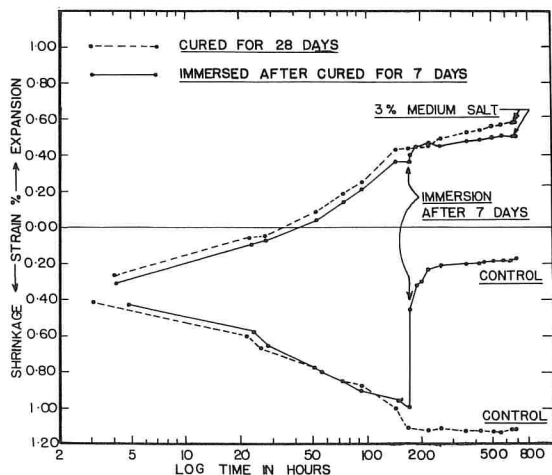
in Figure 12. X-ray diffraction revealed that the moist Ca-montmorillonitic soil-cement mixes with and without salt all initially obtained a 17.7 Å clay basal spacing, approaching the 18.8 Å of the wet clay alone. With the progress of curing, the clay quickly (in a few hours) obtained a stable 14.7 Å basal spacing in specimens with salt with no change in moisture content. On the other hand, in specimens without salt the clay basal spacing decreased very gradually with drying. The results are similar to those shown in Figures 11 and 12. Upon immersion both mixes showed a 001 spacing of about 17 Å.

DISCUSSION OF RESULTS

Shrinkage Mechanism

Figures 11 and 12 suggest that shrinkage in montmorillonitic soil-cement is due mainly to the loss of moisture during curing. This loss causes the clay lattice spacing to decrease, which also contributes an unknown amount of the total shrinkage. The effectiveness of sodium chloride as a shrinkage reducing agent is attributed to the hygroscopic properties of the salt, which, at 3 percent salt content, maintains moisture content in the specimens constant throughout the curing period.

In addition, in Na-montmorillonite the amount of shrinkage that resulted from decreased clay lattice spacing is believed to become less with increasing salt content. This is demonstrated by the fact that the 001 spacing of the Na-montmorillonite in the soil-cement control specimens with 16 percent cement content was found to be 17.7Å soon after wet-mixing, an increase from 11.3Å in the air-dry clay. In the soil-cement specimens with 3 percent fine salt, the spacing was 14.7Å and remained practically constant throughout the curing period (Fig. 11). For 1 and 2 percent fine salt contents, the initial 001 spacings were 17.0Å and 15.0Å respectively. It is believed from these results that the observed increase in shrinkage with decreasing percentages of salt added to the mix was due to the decreasing hygroscopic properties of the mix to maintain the original moisture content and to the weakening abilities of the not-so-plentiful Na^+ cations to prevent calcium saturation of the clay (3, 4, 5) and to achieve lower initial clay basal spacings.

Salt added in solution form is ineffective in reducing shrinkage in Na-montmorillonitic soil-cement (Fig. 7). The reason may be that salt in solution form is more easily going into exchange positions in clay causing expansion of the clay lattice and losing partially its hygroscopic properties to effectively prevent the evaporation of pore waters. X-ray data did show that 0.5 percent salt added in solution form produced an initial Na-clay basal spacing of 17.7 Å, the same as that of the clay in the control soil-cement mix without salt.

Upon immersion, the swelling of Na-montmorillonitic soil-cement without salt (Fig. 13) may be, at least partially, attributed to the increase in clay lattice spacing. X-ray diffraction results did show that the basal spacing returned to 17Å from 14.7Å upon immersion in specimens without salt while the clay in specimens with 3 percent salt maintained at the stable 14.7Å.

In Ca-montmorillonitic soil-cement mixes, the quick reduction of the clay basal spacing from an initial 17.7Å to a stable 14.7Å in specimens with 3 percent salt appears to be due to the exchange of calcium by sodium, as the moisture content remained constant. This quick reduction in clay basal spacing did not produce an apparent effect on shrinkage as the specimens with salt continually showed significantly less shrinkage than the specimens without salt, which, as stated previously, showed gradually decreasing basal spacing with drying throughout the curing period.

Most shrinkage versus time curves (Figs. 1, 2, and 3) show an expanding tendency of salt specimens at longer curing time. X-ray diffraction revealed no sign of recrystallization of salt in the pore spaces, and there was no increase in moisture contents. It is thus speculated that salt might have gradually taken up the exchange position in clay during curing causing dispersing effects of the clay. A dispersed clay structure posseses a larger void ratio and thus results in swelling (8). Similarly, the early shrinkage may be partly attributed to a change to flocculated structures after the dispersing effects of the mechanical molding process.

Effect of Salt Gradations

It was indicated in the previous section that, with a specific salt content, statistical analysis found no significant differences in shrinkage among the various gradations of salt added. However, it is believed that use of a larger number of specimens for analysis would have resulted in greater confidence and, therefore, narrower, nonoverlapping confidence bands. Thus significant differences among some gradations would have been produced, as data shown in Figures 4, 5, and 6 seem to suggest.

Effect of Salt on Strength

The observed reductions in strength in soil-cement specimens with coarser salts are believed to be the result of the salt crystals dissolving, thus leaving "holes" in their places. These holes become areas of stress concentration upon application of load, and the specimen exhibits a lower strength.

CONCLUSIONS

1. Sodium chloride, when used in granular forms, has been shown to be effective in reducing shrinkage in montmorillonitic soil-cement mixtures cured under high relative humidity. The effectiveness increases with increasing salt content and is independent of the gradation of the salt.
2. The reduction of shrinkage in soil-cement with addition of salt was found to be primarily due to the ability of salt to reduce moisture loss in the mixture and to provide a more favorable and stable clay basal spacing. Particle reorientation during curing was also believed to be responsible for some of the shrinkage and expansion observed.
3. Sodium chloride was also found to be effective in decreasing swelling of Na-montmorillonitic soil-cement mixtures upon immersion. A stable, nonswelling clay basal spacing in specimens with 3 percent salt was found responsible.
4. Strength reductions associated with addition of salt to Na-montmorillonitic soil-cement are due only to the coarseness of the additive and not to the amount used. Soil-cement with fine salt added was found to give strength comparable to that of soil-cement specimens without salt.

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Discussion

E. GUY ROBBINS, Portland Cement Association—The authors have presented some very interesting data on the shrinkage of soil-cement with a salt additive. A few general observations are offered for consideration in future research.

The soil used was an active clay mixed with Ottawa sand and a rather high cement factor, 16 percent cement by weight. Salt contents were 1, 2, and 3 percent by weight of materials. This is equivalent to about 6, 12, and 19 percent by weight of cement, which is a high salt content. This suggests soil-cement sodium chloride investigations should be made also with low chloride contents.

In Figures 2 and 6, the mixtures containing 3 percent salt expanded. Figure 10 shows no increase in compressive strength with fine salt and a decrease in 7-day strength with medium and coarse salt. Expansion can indicate some damage to the soil-cement structure, and decrease in strength indicates no improvement in quality. This suggests investigations relating to the durability aspects.

The reduction in shrinkage under moist cure conditions may be due to hygroscopic properties of the salt that could cause the specimens to pick up moisture during curing that would replace moisture lost because of cement hydration, and the net result is less shrinkage. Most shrinkage is associated with loss of moisture.

Volume change studies were made at 90 percent relative humidity. If specimens were dried to, say, 50 percent relative humidity after curing, the volume changes with and without salt admixture may be quite different.

Suggestions for future research include investigations on durability, longer term compressive strength, moisture, and volume change during and after moist curing. Lower salt contents should be investigated with a range of soil types and cement factors.

JERRY W. H. WANG and ALEXANDER H. KREMMYDAS, Closure—The authors wish to thank Mr. Robbins for his interesting discussion and would like to further some of his observations.

It was concluded in the paper that sodium chloride used in granular form was shown to be effective in reducing shrinkage in montmorillonitic soil-cement cured under high relative humidity. For the most part, its effectiveness was due to the hygroscopic properties of salt that make up moisture losses by picking up additional moisture during curing. Inasmuch as NaCl is not capable of absorbing moisture from air at 77 F when relative humidity is below 70 percent, it is anticipated that NaCl would not be very effective in reducing shrinkage at low relative humidity. Nevertheless, the movement of water in the vapor phase and the equivalent relative humidity in a soil-cement mass under normal field conditions are not well understood at this time. Field experimentation is needed to evaluate the limitations of using NaCl as a shrinkage reducing agent in soil-cement.

The magnitudes of critical shrinkage strain that will cause cracking of different soil-cements in the field are also not well known at this time. The high salt contents used in the study were for an active soil showing high shrinkage. It is believed that the amount of salt required to solely prevent cracking can effectively be reduced for most soils and by doing this net expansion will never occur. Again, more studies in the laboratory and in the field are needed in this respect.

The fact that soil-cement with salt would not decrease the strength of soil-cement, provided the coarseness of salt is comparable to the dimensions of the soil-cement mass, is a positive aspect of using salt as a shrinkage reducing agent. In addition, NaCl provides a more favorable and stable clay basal spacing during curing and subsequent immersion for montmorillonitic soil-cement. This fact may offer some practical significance in future soil-cement technology.

Lime Stabilization of Clay-Sand Mixtures

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University of California, Davis

Laboratory-prepared, lime-treated, clay-sand mixtures were studied. A 5 percent lime-treatment level was chosen for the major part of this investigation; however, a limited number of samples were mixed with 3 and 7 percent lime. The percentages of lime specified here were based on the weight of the whole soil mixture. Essentially, 2 types of clay-sand mixtures were investigated: a Hydrite UF-sand mixture and a Grundite-sand mixture. Within each type of mixture, there were different clay-sand ratios. Different compactive efforts were applied to fabricate cylindrical samples of various clay-sand ratios to the desired dry densities, which were taken as 95 percent of the maximum dry density of a specified compactive effort of the corresponding untreated samples. Unconfined compressive strengths and pH values were determined on samples cured for 1 to 12 weeks. The results of this investigation show that the effectiveness of lime-soil stabilization appears to be related to the fine-grain fraction/lime ratio (FGF/L) of a soil mixture. For the 2 types of clay-sand mixtures studied, the optimum FGF/L ratios for maximum strength range from 14 to 12 depending on curing period. The rate of strength gain is affected by the lime-treatment level; however, the optimum FGF/L ratio is only slightly affected. The magnitudes of maximum strength for a given FGF/L ratio vary with the coarse-grain fraction content in a soil mixture—the lower the coarse-grain fraction content is, the higher the strength. It is believed that the FGF/L ratio is a more indicative parameter for dealing with lime-soil stabilization than the lime percentage specified on the basis of either total weight or total volume of the whole soil mixture.

• LIME HAS BEEN USED extensively to modify the engineering characteristics of fine-grained soils. The beneficial effects of lime stabilization on the plasticity, shrinkage, workability, and strength properties of a soil are well known. In general, most of these properties are altered by the addition of 3 to 7 percent lime by weight. The strength increase observed in soils by the addition of lime, however, is variable. This variation of strength increase has been attributed to the degree of accomplishment of lime-soil reactions, namely, cation exchange, flocculation, carbonation, and pozzolanic reaction. The last reaction is considered to be primarily responsible for the long-term strength increase in lime-soil mixtures.

The effectiveness of lime stabilization depends on many factors. Works by groups from M.I.T., Iowa State University, University of Illinois, and many others have contributed greatly to the understanding of the engineering behavior, the chemical reactions, and the mineralogical aspect of lime-soil stabilization. In recent years, a qualitative approach based on pedological classification has been adopted by Thompson [1] to establish some guidelines for the evaluation of lime reactivity of Illinois soils. He reported that lime-soil reactions are dependent on the nature and character of the soil being stabilized. Hilt and Davidson [2] reported that lime fixation in clayey soils depends on

the type of clay minerals. The quantity of lime used for lime fixation contributes to the improvement of soil workability but not to the increase in strength. Additional amounts of lime added above the lime fixation capacity cause the formation of cementing materials within clayey soils.

X-ray diffraction and electron microscopic studies made on lime-soil stabilization (3, 4, 5) have indicated that pozzolanic reaction can be described as a slow and contin- uous reaction in the presence of $Ca(OH)_2$ and soluble silica or alumina. It causes the breakdown of clay particles and the formation of new crystalline phases, or it attacks the clay particles and deteriorates the whole clay mineral structure. Because of these processes the conductivity of the system decreases, indicating that soluble salts in the lime-soil mixture are converted to less soluble compounds that may serve to bond par- ticles together. This phenomenon is irreversible.

Eades and Grim (6) more recently have proposed that the optimum lime content for strength increase for lime stabilization can be determined by pH readings of the lime- soil mixture. Hilt and Davidson (2) reported that the lime fixation capacity of a mont- morillonitic or kaolinitic soil is the same as the optimum lime additive for maximum increase in the plastic limit of the soil. Arulanandan and Shen (7) have used the non- destructive electrical response characteristics measurements to monitor the continuous structural change of a lime-soil mixture. This technique makes possible the examina- tion of the various lime-soil reactions and the determination of the approximate lime percentage required for long-term strength increase.

It is interesting to note that most of the work done in lime-soil stabilization specifies the amount of lime added to the mixture as a percentage of either the total weight or the total volume of the soil mixture. However, it is generally recognized that lime reacts primarily with the fine-grain fraction (passing No. 200 sieve) of the soil mixture, whereas the coarse-grain fraction does not react chemically with lime. Therefore, it is worthwhile to specify the lime content for stabilization on the basis of the fine-grain fraction of the soil mixture rather than the mixture as a whole and relate the effective- ness of lime stabilization to the ratio of fine-grain fraction to lime content. It would also be interesting to examine how the chemically nonreactive part of the soil mixture affects the overall physical properties of lime stabilization. This approach appears to be more realistic in dealing with natural soils that are in most cases composed of var- ious amounts of sand, silt, and clay.

This paper presents the results of a preliminary study made on laboratory mixed clay-sand mixtures treated with lime.

EXPERIMENTAL PROGRAM

Materials

The sand used in this study was a No. 20 Del Monte sand of uniform subrounded to subangular particles. The gradation curve of this sand is shown in Figure 1. Also shown in Figure 1 are the grain-size distribution curves of the 2 types of commercial clays used in this study. They are Hydrite UF, a product from George Kaolin Company, and Grundite, a product from Illinois Clay Products Company. The Hydrite UF is a pure kaolin clay of very fine particles, and the Grundite contains primarily illitic clay minerals with substantial amounts of silt-size particles. The physical properties of these commercial clays are given in Table 1. A hydrated, high-calcium lime contain- ing 90 percent available $Ca(OH)_2$ was used in all the mixtures.

Lime-Treatment Level

A 5 percent lime-treatment level was chosen for the major part of this investigation; however, a limited number of samples were treated with 3 and 7 percent lime. The percentages of lime specified here were based on the weight of the whole soil mixture.

Preparation of Specimens

Composition of Specimens—Figure 2 shows the compositions of the specimens fab- ricated and tested in this investigation. Essentially, there were 2 types of mixtures:
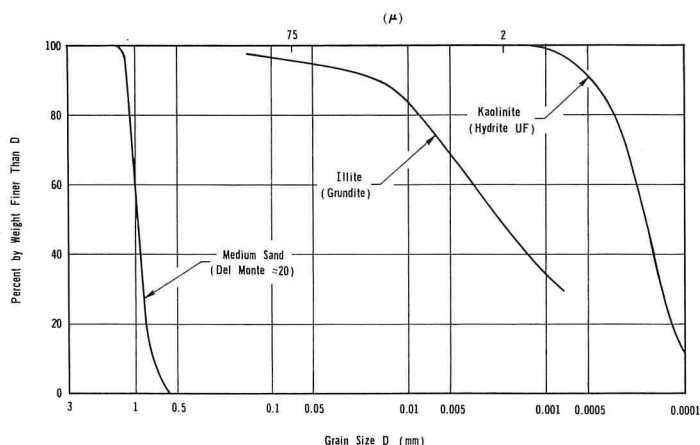
Figure 1. Grain size distribution curves for experimental clays and sand.

a Hydrite UF-sand mixture (K-S), and a Grundite-sand mixture (I-S). Samples were labeled as, for instance, 5K100, which meant 100 percent Hydrite UF and no sand treated with 5 percent lime, or 3G60, which meant 60 percent Grundite and 40 percent sand treated with 3 percent lime.

Mixing—The appropriate amount of clay, sand, and lime were first mixed in an air-dry condition; the necessary amount of water was then added to the mixture and thoroughly mixed for about 5 minutes. The time lapse between mixing and compaction was kept constant for all samples. The amount of water-soil-lime mixture mixed each time was enough for 2 specimens, and the time lapse from after mixing to the completion of compaction was approximately 15 minutes.

Compaction—Cylindrical samples were compacted in pairs by static compaction in 1.4-in. diameter steel molds. Different compactive efforts were employed to fabricate samples of various clay-sand ratios to the desired dry densities, which were

TABLE 1

CHARACTERISTICS OF COMMERCIAL CLAYS

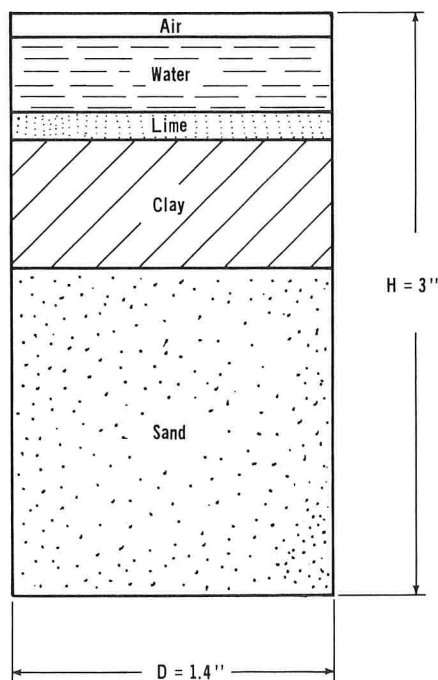| Characteristic	Kaolinite (Hydrite UF)	Illite (Grundite)
Liquid limit	63	51
Plasticity index	34	30
Percentage finer than No. 200 sieve	100	95
Percentage finer than 2μ	100	47
Mineral composition, percent	100 Kaolinite	55 Illite (9)
		10 Kaolinite
		20 Quartz
		15 Mixed layer clay

Figure 2. Composition of a specimen.

taken as 95 percent of the maximum dry density of a specified compactive effort of the corresponding untreated samples. The molding moisture contents were taken as 112, 120, and 128 percent of the optimum moisture contents of the untreated samples for 3, 5, and 7 percent lime-treatment levels respectively. The maximum dry densities and the optimum moisture contents of the untreated samples are given in Table 2.

Curing—All samples were stored in a moisture room of 72 F and 95 percent humidity for curing. These samples were wrapped in 2 rubber membranes with a thin film of silicon grease in between. O-ring seals were applied around both lucite bases to prevent change in moisture content and the entry of CO_2 gas during curing.

The 5 percent lime-treatment samples were cured for 1, 2, 4, 8, and 12 weeks prior to testing. A 4-week curing period was chosen for I-S mixtures of 3 and 7 percent lime treatment. The untreated samples were also cured for one week before testing to eliminate possible thixotropic effect in compacted clay.

Testing

Unconfined Compression Tests—The unconfined compression tests were carried out on the TO testing machine. The loads and deformations throughout the test were automatically recorded. All tests were performed at a strain rate of 0.05 in. per minute.

pH Value Measurements—The Backman pH-meter was used to measure the pH of all samples. As recommended by Eades and Grim (6), a 1:5 soil to CO_2-free distilled water slurry was used. The slurry was stirred at regular intervals for one hour before measurements were taken.

TABLE 2

MAXIMUM DRY DENSITIES AND OPTIMUM MOISTURE CONTENTS OF UNTREATED MIXTURES

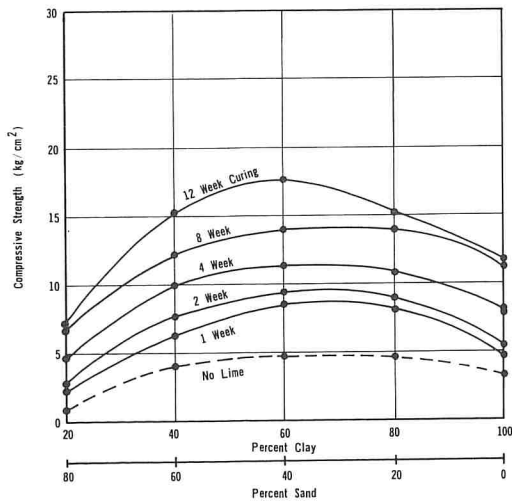| Mixture	Dry Density (g/cm^2)	Moisture Content (percent)
K 100	1.27	36.0
K 80	1.42	26.5
K 60	1.61	21.0
K 40	1.82	15.2
K 20	1.97	10.5
I 100	1.66	20.0
I 80	1.76	17.0
I 60	1.85	14.5
I 40	1.98	12.5
I 20	1.98	10.0

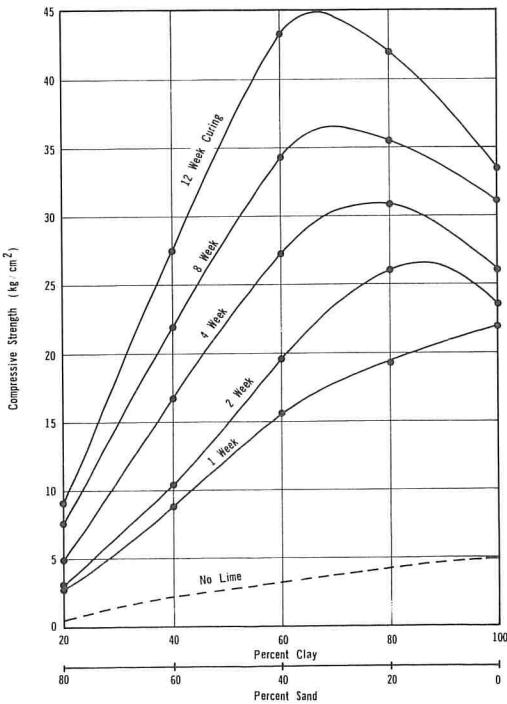Figure 3. Compressive strength of kaolinite-sand mixtures with 5 percent lime.

Figure 4. Compressive strength of illite-sand mixtures with 5 percent lime.

TABLE 3
UNCONFINED COMPRESSIVE STRENGTH DATA (kg/cm²)

Specimen	Untreated	5 Percent Lime Treatment				
		1W	2W	4W	8W	12W
K 20	1.0	2.4	2.5	4.7	6.8	7.1
K 40	4.1	6.2	7.6	10.1	12.2	15.1
K 60	4.8	8.6	9.4	11.0	13.9	17.9
K 80	4.7	8.1	8.9	11.0	14.6	15.0
K 100	3.3	4.7	5.4	8.2	11.5	11.3
I 20	0.6	2.8	3.1	4.9	7.6	9.0
I 40	2.3	8.9	10.6	16.7	21.9	27.6
I 60	3.0	16.1	19.7	27.1	34.4	43.5
I 80	4.4	19.3	26.1	30.9	35.5	42.0
I 100	5.2	22.0	23.5	26.0	31.0	33.3

TEST RESULTS

Strength Tests

After a predetermined period of curing, samples were tested to failure, and the unconfined compressive strengths of the samples were determined. The results of strength tests plotted against the sand-clay ratio for K-S and I-S mixtures are shown in Figures 3 and 4 respectively. The dotted curve on the lower part of each plot shows the compacted, untreated soil strengths of different C/S ratios. It can readily be seen that lime treatment can increase the strengths of the mixtures (compared with 12-week curing strength at same densities) from 4 to 7 times their respective untreated strengths for K-S mixtures and about 6 to 16 times for I-S mixtures. The strength test results are given in Table 3.

Figures 5 and 6 show the variations of axial strains at failure for the 5 percent lime-treatment samples. These results indicate that in general the strain at failure decreases with increasing curing time and decreasing fine-grain fraction content in a sample.
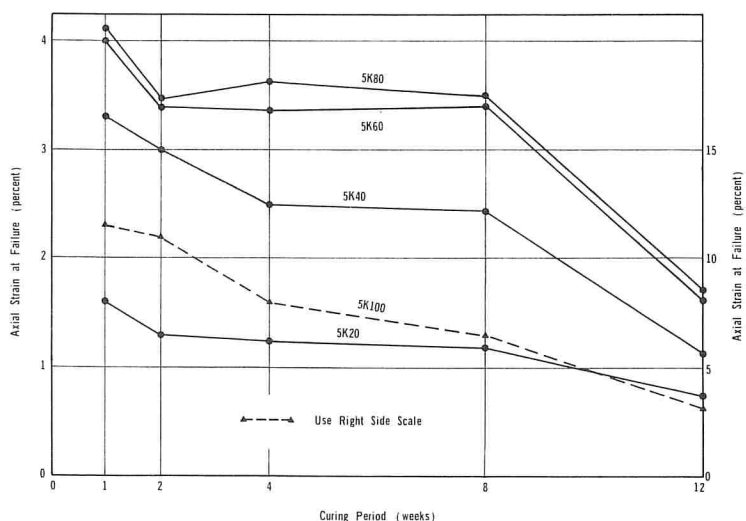

Figure 5. Axial strain at failure of kaolinite-sand mixtures with 5 percent lime.
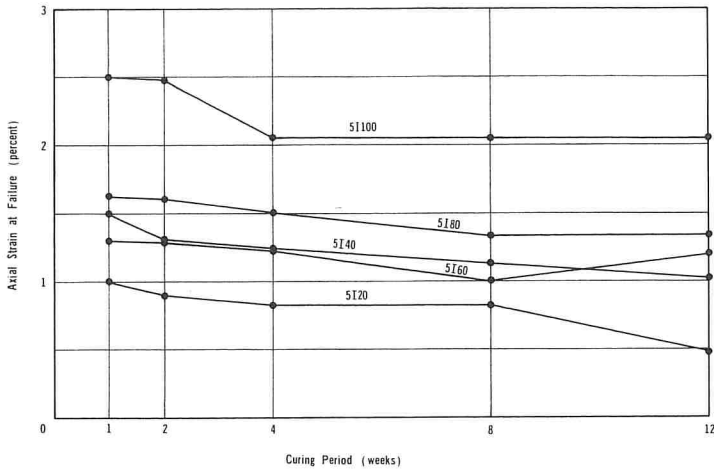
Figure 6. Axial strain at failure of illite-sand mixtures with 5 percent lime.

pH Measurements

Upon completion of the unconfined compression tests, samples were broken up partly for water content determination and partly for pH value measurements. The pH value of a soil sample was measured with 5 parts of CO_2-free distilled water, and 1 part of the soil mixture. Figures 7 and 8 show the variations of pH values for various C/S mixtures with curing time. The untreated mixtures had much lower pH values (Table 4).
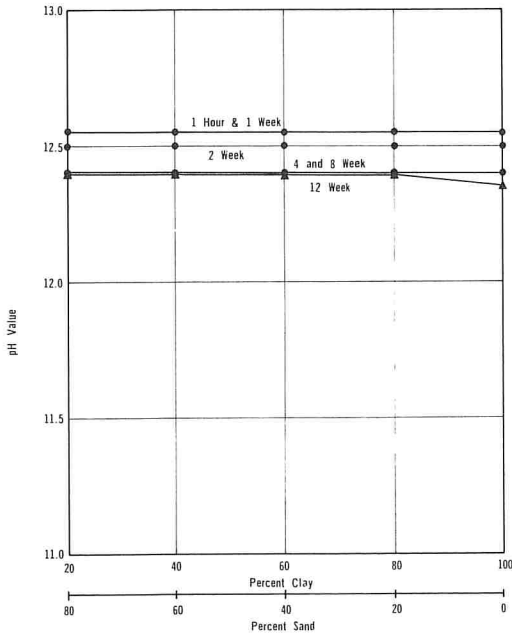

Figure 7. pH values of kaolinite-sand mixtures with 5 percent lime.
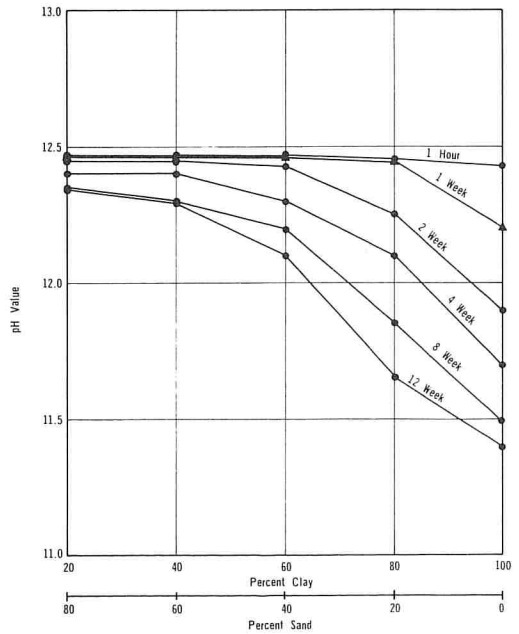
Figure 8. pH values of illite-sand mixtures with 5 percent lime.

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TABLE 4

pH VALUES

</div>

Specimen	Untreated	5 Percent Lime					
		1H	1W	2W	4W	8W	12W
K 20	5.2	12.55	12.55	12.50	12.40	12.40	12.40
K 40	4.9	12.55	12.55	12.50	12.40	12.40	12.40
K 60	4.9	12.55	12.55	12.50	12.40	12.40	12.40
K 80	4.9	12.55	12.55	12.50	12.40	12.40	12.40
K 100	4.9	12.55	12.55	12.50	12.40	12.40	12.35
I 20	3.5	21.47	12.20	11.90	11.70	11.50	12.35
I 40	3.2	12.47	12.45	12.25	21.10	11.85	12.30
I 60	3.0	12.47	12.47	12.43	12.30	12.20	12.10
I 80	3.0	12.45	12.47	12.45	12.40	12.30	11.65
I 100	2.9	12.43	12.47	12.45	12.40	12.35	11.40

The addition of 5 percent lime to the soil mixture immediately elevated the pH values to approximately 12.5 for all mixtures. The pH value decreased as curing time was prolonged. However, the amount of decrease in pH with time depends on the C/S ratio and the clay mineral of the mixture. The relatively rapid drop in pH in I-S mixtures reflects the higher strength-gaining capacity of the mixtures.

Lime-Treatment Level

A limited number of samples of I-S mixtures were prepared with 3 and 7 percent lime. These samples were cured for 4 weeks prior to testing. These tests were designed to investigate the effect of lime-treatment level on the strength of samples having various C/S ratios. The results of these tests are shown in Figure 9. pH values were also determined after the completion of strength tests and are shown in Figure 10.

DISCUSSION OF RESULTS

Compressive Strength

K-S Mixtures—For samples with a fine-grain fraction greater than 40 percent as shown in Figure 3, the strengths of untreated samples range from 3.5 to 4.5 kg/cm^2. For samples with a fine-grain fraction of less than 40 percent, the untreated strength decreases drastically
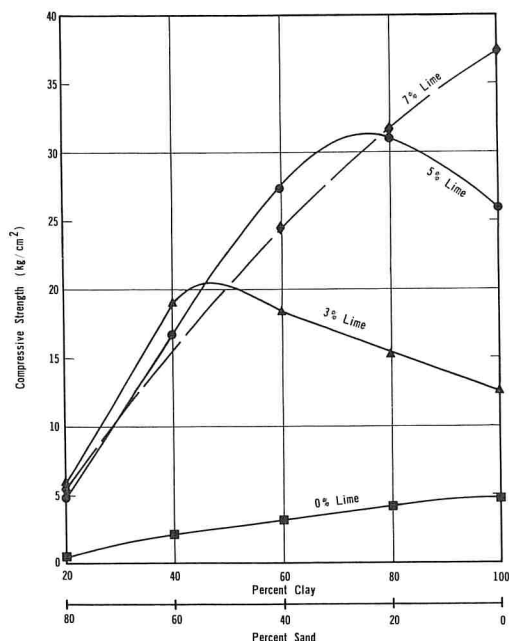

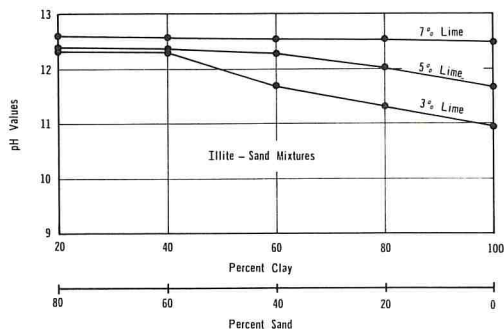Figure 9. Illite-sand mixture with 4-week curing strength.

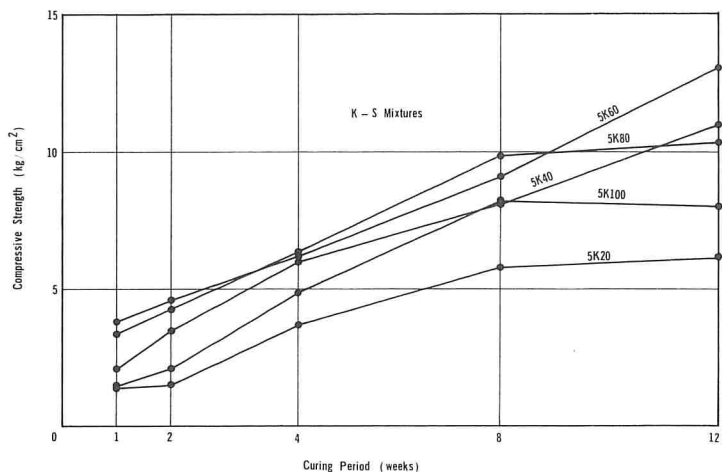Figure 10. pH variations with lime-treatment level after 4-week curing.

Figure 11. The increase in compressive strength with curing period of K-S
mixtures with 5 percent lime.

because of lack of cohesion. The addition of 5 percent lime increases the strengths of
all samples; in general, the longer the curing period is, the stronger the sample. The
percentage of strength increase is the greatest in the low fine-grain fraction range.
This results from the fact that the untreated strengths of those samples are extremely
low. The rate of strength gain due to pozzolanic reaction, however, is not the same
for samples of different fine-grain fractions. For high strength gain the most effective
range is between 40 and 80 percent depending on the curing period (1 to 12 weeks). Fig-
ure 11 shows the strength increase with time for different C/S ratios, indicating that the
rate of strength increase varies with both the curing period and the fine-grain fraction
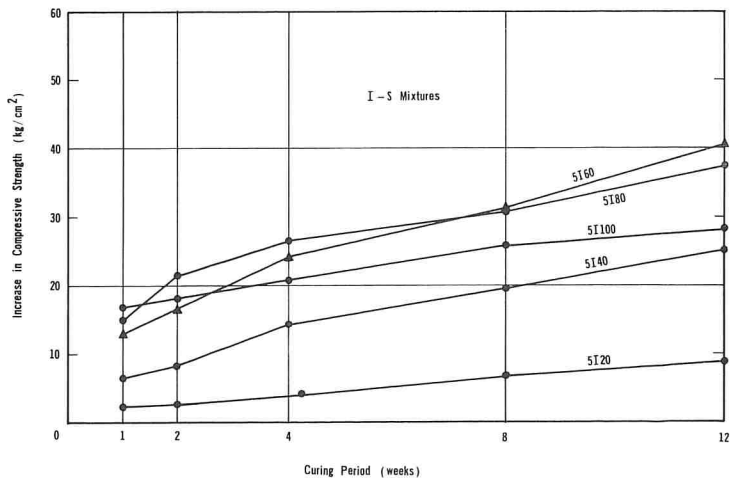content.

Figure 12. The increase in compressive strength with curing period of I-S
mixtures with 5 percent lime.

TABLE 5

FINE-GRAIN FRACTION/LIME RATIO OF CLAY-SAND MIXTURES

Specimen	3 Percent Lime	5 Percent Lime	7 Percent Lime
I 20	6.4	3.8	2.7
I 40	12.7	7.6	5.4
I 60	19.0	11.4	8.2
I 80	25.4	15.2	10.9
I 100	31.7	19.0	13.6
K 20		4.0	
K 40		8.0	
K 60		12.0	
K 80		16.0	
K 100		20.0	

I-S Mixtures—The same tendency of strength variation can also be seen in I-S mixtures as shown in Figures 4 and 12. However, the rate and magnitude of strength gain are much more significant, indicating the high lime reactive nature of the fine-grained soil. The most effective range for high strength gain in this case is between 60 and 100 percent and, like the K-S mixtures, the maximum strengths move from samples of high to low fine-grain fraction content as the curing time is prolonged. Data presented by McDowell (8) have shown that the optimum lime content for maximum strength is not a constant but rather it varies with curing time for any given soil.

Fine-Grain Fraction/Lime Ratio

The bulk of this test program was based on samples treated with 5 percent lime by weight of the total soil mixture. Because of the various C/S ratios of the samples, the fine-grain fraction/lime ratios of these samples were different and are given in Table 5.

The gain of strength of lime-stabilized soils is regarded primarily as a result of pozzolanic reaction between soil silica and/or alumina and lime to form various types of cementing agents. The possible sources of silica and alumina in soils are clay minerals, quartz, feldspars, micas, and other similar very fine silicate or aluminosilicate minerals (1). The clay fraction of a soil is generally considered as the major source of silica or alumina, or both, for the lime-soil pozzolanic reaction; however, minerals of the silt fraction may also serve as a source for pozzolanic reaction. Thompson (1) has reported that the clay content of a soil is not indicative of its lime reactivity, and soils having low clay content can be adequately stabilized with 3 to 7 percent lime. It appears proper, then, to use fine-grain fraction/lime ratio to define the effectiveness of lime-soil stabilization.

The optimum FGF/L ratios for maximum strength of various curing periods are shown in Figure 13 for both K-S and I-S mixtures. The optimum ratios for K-S mixtures vary within a rather narrow range (14 to 12), whereas for I-S mixtures this ratio decreases rapidly from 1 week to 4 weeks curing (19 to 14.3); however, the change of optimum FGF/L ratio from 4 to 12 weeks is gradual and slow (14.3 to 12.4). It is generally recognized that the effectiveness of lime stabilization is measured by the 4-week or longer curing strength; therefore, it is reasonable to conclude that the optimum FGF/L ratios for both mixtures studied are similar, ranging from 14 to 12 depending on the curing period. It appears then that the
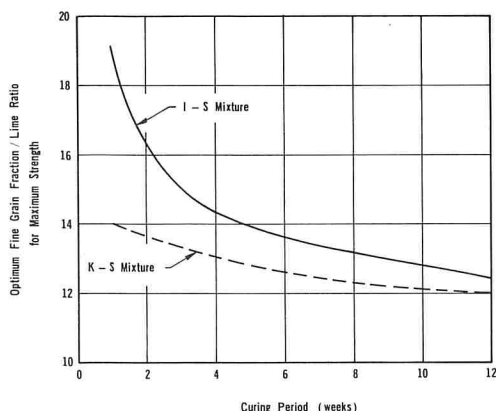

Figure 13. The variation of optimum fine-grain fraction/lime with curing period for mixtures with 5 percent lime.

optimum amount of lime needed for any soil mixture can be determined by the gradation curve of the mixture and the proper range of FGF/L ratio. This ratio according to the present study ranges from approximately 12 to 14. The conventional method of determining optimum lime content for any soil mixture can thus be simplified.

Lime-Treatment Level

The 3 and 7 percent lime-treated I-S samples were compared with those with 5 percent lime treatment of the same curing age. Figure 14 shows that the rate of strength gain is affected by the lime-treatment level. After 4 weeks of curing, the maximum strength of the 7 percent lime samples was 7I100 (optimum FGF/L = 13.6), that of 5 percent lime samples was between 5I60 and 5I80 ($\approx$ 14.5), and that of 3 percent lime samples was between 3I40 and 3I60 ($\approx$ 15). It is important to note that the optimum FGF/L ratios are very close even though the compositions and lime contents of these samples are quite different. This indicates that the optimum FGF/L is probably unaffected by the lime-treatment level.

Figure 14. The increase in compressive strength with various lime content and 4-week curing period.

Coarse Grain Fraction Content

The strengths of samples having the same FGF/L ratios are affected by the amount of coarse-grained soil contained in the samples. Figure 15 shows the 4-week curing strengths of I-S mixtures with 3, 5, and 7 percent lime; generally speaking, the lower the coarse-grained soil content is, the higher the strength for a given FGF/L ratio. This difference in strength is due to the different amount of available silica, alumina, and lime in the mixture for lime-soil pozzolanic reaction. Although the optimum FGF/L is probably unaffected by the lime-treatment level for the 2 types of minerals used in this study, the magnitudes of maximum strength for any given FGF/L ratio do vary with the coarse-grained soil content of a sample.
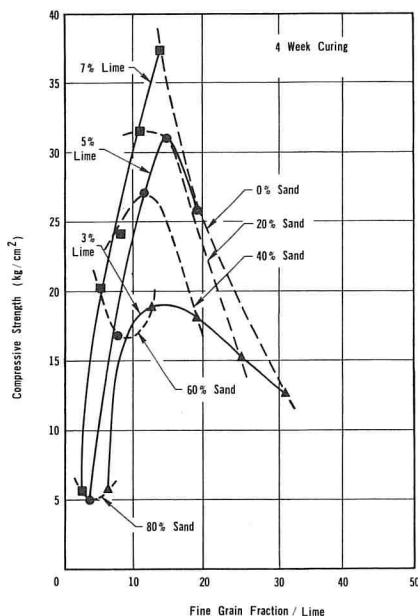

Figure 15. Relationships of compressive strength, fine-grain fraction/lime ratio, and percentage of sand content.
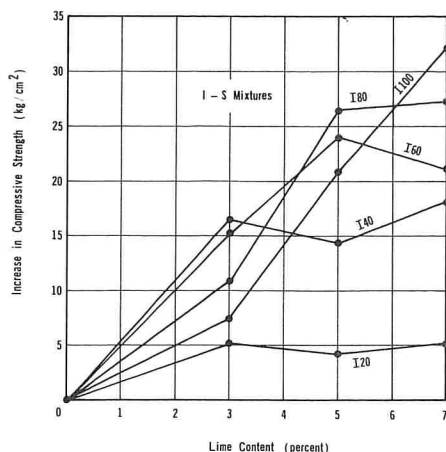
CONCLUSIONS

This study was conducted in the laboratory on lime-stabilized samples of various clay-sand mixtures. These samples were compacted to the same densities as the corresponding untreated samples. Conclusions from this study may be summarized as follows:

1. Comparison of untreated and 12-week curing samples indicates that the addition of 5 percent lime to various K-S and I-S mixtures causes an increase in compressive strength from 4 to 7 times for K-S mixtures and 6 to 16 times for I-S mixtures.

2. The pH value of the lime-soil mixtures decreases with longer curing period and larger fine-grain fraction content of a sample. However, no significant correlation between strength gain and pH variation can be established.

3. The Grundite soil that contains primarily illitic clay mineral and a large amount of silt-size particles reacts better with lime than the Hydrite-UF soil of very fine pure kaolin particles.

4. The effectiveness of lime-soil stabilization appears to be related to the FGF/L ratio of a soil mixture. For the 2 types of clay-sand mixtures studied, the optimum FGF/L ratios for maximum strength range from 14 to 12 depending on curing period.

5. The FGF/L ratio is a more indicative parameter for dealing with lime-soil stabilization than the lime percentage specified on the basis of either total weight or total volume of the whole soil mixture.

6. The rate of strength gain is affected by the lime-treatment level; however, the optimum FGF/L ratios for maximum compressive strengths is only slightly affected.

7. The magnitudes of maximum strength for a given FGF/L ratio vary with the coarse-grained soil content in a mixture; the lower the coarse-grained soil content is, the higher the strength.

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Effects of Sodium Chloride and Sodium Chloride-Lime Admixtures on Cohesive Oklahoma Soils

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Sodium chloride has been used for many years as a stabilizing admixture in "select" base course material. However, little if any investigation of the engineering properties of sodium chloride-stabilized cohesive soil has been done. This paper describes the effects of sodium chloride and sodium chloride-lime admixtures on plasticity, compaction, and strength properties of 2 high-volume-change cohesive Oklahoma soils. After review of available literature and discussion of chemical and physical reactions that occur during stabilization, experimental test results are presented and discussed. Small percentages of sodium chloride added to raw soil were found to have negligible effects on soil plasticity while increasing compacted density and decreasing optimum compaction moisture content. Workability and moisture-retention qualities of the raw soil were also enhanced. When small percentages of sodium chloride were added to lime-modified soil, similar results were obtained: Compacted density was increased, optimum moisture decreased, and the workability and moisture-retention properties of the salt-lime mixtures were enhanced considerably over those of lime-modified soil. In addition, salt-lime treatment produced strength gains over those obtained by lime treatment alone, both at the maximum compacted density and optimum moisture for each treatment and when the soils were compacted to similar moisture and density conditions. Reasons for the behavior observed are presented.

•CHEMICAL TREATMENT of subgrade soils to produce more desirable foundation material has gained increasing attention in recent years. Lime, in both hydrated and oxide form, has become one of the most widely used chemical treatments. Although lime treatment produces very desirable results, some undesirable effects also occur. These effects are more evident in lime modification than in stabilization and include reduced compacted unit weight, increased optimum moisture content, and little change in workability during initial mixing operations.

Addition of other chemical additives in conjunction with lime might be an effective way to minimize undesirable effects. The School of Civil Engineering at Oklahoma State University, Stillwater, is presently engaged in a feasibility study to determine effects of sodium chloride and sodium chloride-lime admixtures on cohesive Oklahoma soils. The project is sponsored by the Oklahoma Department of Highways.

This paper presents preliminary results obtained in the study. Theoretical considerations concerning the effect of lime and salt on cohesive soils are reviewed, followed by presentation and discussion of experimental results.

THEORETICAL CONSIDERATIONS

Addition of lime to cohesive soil usually produces a decrease in plasticity. Chemical reactions that occur during lime treatment are not completely understood but must be hypothesized to explain effects of lime on cohesive soil. As divalent calcium ions are released on addition of lime to cohesive soil, monovalent diffused ions in the double water layer are dissociated. This causes some compression of the double layer and thus flocculation of clay particles. At the point of complete double-water-layer compression, no further reduction in plasticity is possible because the clay surface charge has been brought to equilibrium. The lime content that produces this maximum plastic limit has been termed the "lime-fixation point" by some investigators (1) and throughout this paper will be referred to as "modification optimum," for at this lime content, maximum modification of soil with respect to plasticity reduction has been obtained.

Addition of lime above modification optimum provides more free calcium ions, which are available for formation of new molecules and crystals. Calcium combines with aluminum hydroxyl groups to form tetracalcium aluminate hydrates that produce quick, low-strength cementing bonds within treated soil (2). Higher, long-term strength gains are obtained from formation of tobermorite minerals. These minerals are strongly bound hydrates of calcium and silica. Formation of tobermorites, such as calcium silicate hydrate, by pozzolanic reaction has been found to be greatly enhanced under high pH conditions. A pH in excess of 10.0 increases the solubility of silica, thus acting as a catalyst for new mineral formation (3).

Many factors exist that affect behavior of lime-treated cohesive soils. The effect of particular cations present in soil has been discussed many times; however, because formation of new crystals depends on availability of high valence hydrates, it seems only reasonable to conclude that soil sesquioxide content may be very important in lime reactions. Mattson (4), in some of his early work, found that silica/sesquioxide ratios in clays governed the behavior of these soils in reactions with acids and salts. Because free silica is all-important in the formation of tobermorites, it appears that lime treatment may be more dependent on the soil silica/sesquioxide ratio than any other single factor, as most lateritic soils, having low silica/sesquioxide ratios, react very poorly to lime treatment.

Addition of sodium chloride to cohesive soil has the same effect as addition of any other neutral salt. Phenomena that occur upon treatment of clay with salt may be explained in terms of double-water-layer theory or molecular chemistry. Both approaches produce the same result—a reduction in pH of the soil water system.

Clay particle surfaces are negatively charged, with net charge decreasing as distance from the particle surface decreases. This phenomenon is a result of diffused cations existing in the diffused double water layer. As salt is added to soil, double-water-layer thickness is reduced as a result of concentration of diffused cations. Compression of the diffused double layer causes a more abrupt decrease in charge across the layer, thus reducing the pH of the entire system (5).

Chemically, salt added to soil reacts with aluminum and silica in clay minerals to the extent that the minerals are altered. Addition of salt to clay minerals produces an excess of aluminum ions, which react with anions to form aluminum salts, very acidic compounds (6). The increased solubility of silica in the presence of sodium chloride, as shown by Van Lier, DeBruyn, and Overbeek (7), would enhance aluminum bonding that produces soil acidity.

MATERIALS AND SAMPLE PREPARATION

Materials being used in the study are 2 cohesive soils native to Oklahoma. The first type is a red permian clay (PRC) obtained from a depth of approximately 10 ft- on the campus of the Oklahoma State University, Stillwater. The second cohesive soil is a highly plastic gray clay (RMGC) obtained from Roger Mills County in western Oklahoma. The gray clay deposit was found at a depth of approximately 3 ft, above permian deposits in that area. Index properties of PRC and RMGC are given in Table 1. Grain size distribution curves for both clays are shown in Figure 1. It should be noted that RMGC is more plastic than PRC, despite a lower colloidal fraction.

Quicklime used in the study was obtained from the St. Clare Lime Company, Sallisaw, Oklahoma. Both commercial grade sodium chloride and ordinary rock salt were used in the study, without any noticeable effect on test results. A standard mixing and curing procedure was adopted for all samples prepared, and it was found to produce mixtures with more nearly constant moisture contents and better workability than any other procedure tried. Soil and additives were thoroughly mixed in dry form. Required moisture contents were obtained by sprinkling the entire sample surface with water. Mixtures were then sealed and allowed to cure for 8 to 12 hours at room temperature. This allowed moisture to migrate evenly and naturally through the soil samples.

TABLE 1

INDEX PROPERTIES OF PRC AND RMGC

Properties	PRC	RMGC
Specific gravity	2.72	2.73
Liquid limit	38.6	60.5
Plastic limit	17.6	29.8
Plasticity index	21.0	30.7
Flow index	3.0	7.7
Toughness index	7.0	4.0
Liquidity index	—	0.33
Lineal shrinkage, percent	12.0	17.8

PRESENTATION AND DISCUSSION OF RESULTS

Primary objectives of the investigation involved effects of sodium chloride and lime admixtures on engineering behavior of cohesive soils; however, it was necessary to determine effects of individual additives on the clays for comparative purposes. In the following sections the effects of salt treatment, lime treatment, and salt-lime treatment on the 2 cohesive soils are shown and discussed.

Effect of Sodium Chloride Admixtures on Soil Plasticity and Compaction

Addition of sodium chloride to both soils had the same general effect on plasticity, being more pronounced in RMGC. Increased percentages of salt caused corresponding increases in soil liquid limits (Fig. 2). A general increase was found to occur in plastic limit values for PRC; however, RMGC had a maximum plastic limit at 1 percent salt content. Plasticity index values for both soils increased with increasing salt content. The
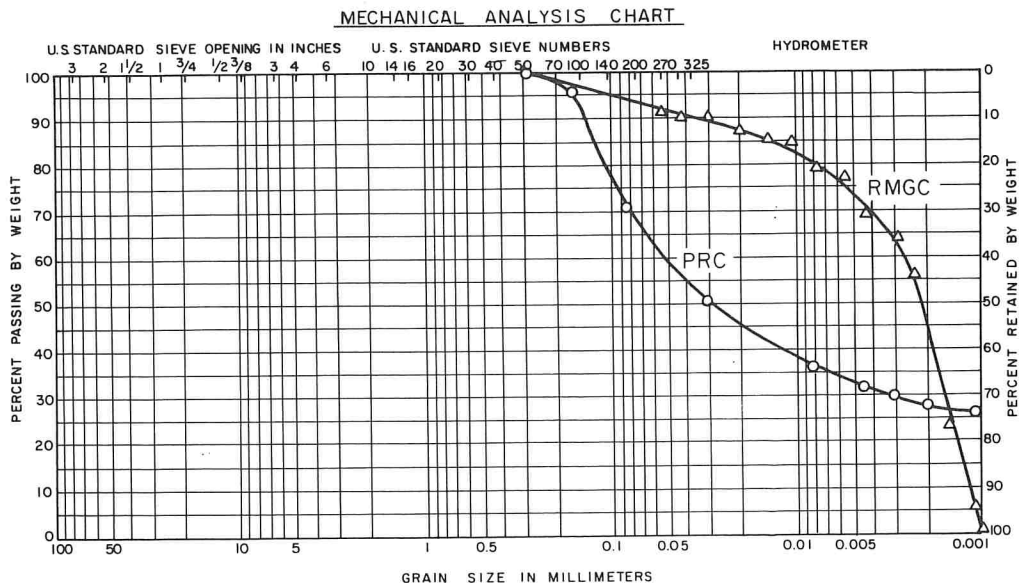

Figure 1. Grain size distribution curves for PRC and RMGC.

maximum increase in PI of PRC was 4.0 while RMGC showed an increase of 14.0, both at 4 percent salt. At salt contents below 2 percent, the effect of salt treatment on plasticity was negligible.

From observed behavior of these widely different soils, it appears that surface chemistry had a large effect on behavior. PRC, high in sesquioxide content, seems to be more stable on treatment with salt than does RMGC. This is reasonable because monovalent sodium ions would have little effect on dissociation of trivalent ions existing near PRC particle surfaces. However, salt treatment would have a greater effect on RMGC because of its low sesquioxide content and the absence of large amounts of high-valence ions near clay surfaces.

Standard Proctor density tests conducted on sodium chloride-treated soil produced favorable results in the case of PRC. Compacted unit weight of PRC was increased with increase in salt content to a maximum of 112 lb/cu ft at 2 percent salt, an increase of 6.0 lb/cu ft over that for the raw soil. This gain is much greater than that contributed by the presence of sodium chloride. RMGC produced no gain in compacted unit weight with addition of salt. Corresponding optimum moisture contents were found to

Figure 2. Effect of salt treatment on Atterberg limits of PRC and RMGC.

be reduced with increase in salt content, passing through a minimum at 2 percent salt. Optimum moisture for PRC decreased from 18.0 percent in raw soil to 15.0 percent with addition of 2 percent salt. RMGC showed no reduction in optimum moisture content with salt treatment.

Because moisture-density curves obtained during compaction tests depend primarily on particle orientation, it would be reasonable to expect that the flocculating effect of salt affected particle orientation. The authors feel that, for PRC, 2 percent salt produces enough compression of the double water layer to obtain maximum compacted density at the lowest possible moisture content. However, compacted density of RMGC was not affected by salt treatment. This could result from differences in mineral composition, as PRC has a larger colloidal fraction even though RMGC is more plastic.

Quantitatively, moisture retention of salt-treated soils of both types was much greater than for raw soil samples. This was evident in the mixing and curing procedures described previously.

Effect of Lime Admixtures on Soil Plasticity and Compaction

Atterberg limits were run on admixtures of both PRC and RMGC with varying percentages of lime, in order to locate the modification optimum lime content. Modification optima for PRC and RMGC were 4 and 6 percent respectively. To further substantiate location of the modification optimum, the pH test for lime-treated soil was used to locate the optimal lime content, and confirmed the Atterberg limit-obtained percentages.

Standard Proctor density data for the soils at their respective lime modification optima indicated a relatively large reduction in compacted unit dry weight from that of the raw soil, with resulting increased optimum compaction moisture content. Maximum compacted unit dry weight for PRC was decreased from 106 to 95 lb/cu ft by addition of 4 percent lime, while optimum moisture increased from 18.0 to 20.5 percent. For RMGC, 6 percent lime decreased maximum compacted unit dry weight from 96 to 91 lb/cu ft, and optimum moisture increased from 20.0 to 26.5 percent.
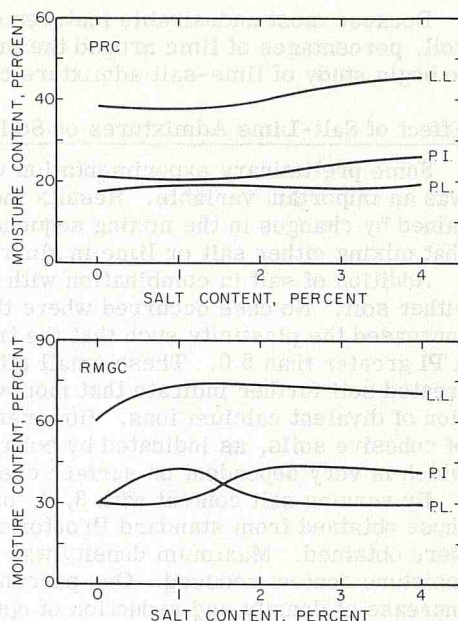
Because most undesirable features of lime treatment occur in lime modification of soil, percentages of lime around the modification optimum of each soil were selected to begin study of lime-salt admixture combinations.

Effect of Salt-Lime Admixtures on Soil Plasticity and Compaction

Some preliminary experimentation was conducted to determine if mixing procedure was an important variable. Results indicated that no appreciable difference was obtained by changes in the mixing sequence of salt and lime, nor was there any evidence that mixing either salt or lime in slurry form had any significant effect on soil behavior.

Addition of salt in combination with lime had very little effect on the plasticity of either soil. No case occurred where the addition of small percentages of salt with lime increased the plasticity such that the treated soil became nonselect material, i.e., with a PI greater than 5.0. These small effects of sodium chloride on plasticity of lime-treated soil further indicate that monovalent sodium ions have little effect on dissociation of divalent calcium ions. However, sodium chloride does affect surface chemistry of cohesive soils, as indicated by behavior of salt-lime mixtures during compaction, which is very dependent on surface chemistry and particle orientation.

By varying salt content with 3, 4, or 5 percent lime plus PRC, trends similar to those obtained from standard Proctor compaction tests of PRC plus sodium chloride were obtained. Maximum density was found to be substantially increased, and optimum moisture content reduced. One percent salt appeared to be the optimal salt content for increase of density and reduction of optimum moisture content in lime-modified PRC. Similar results were found to occur with RMGC, although response to the salt-lime treatment was not as pronounced as with PRC. An optimum salt content of 2 percent was found for RMGC mixtures. Results obtained from standard Proctor compaction tests for lime contents at and near modification optimum are shown in Figure 3. Effects of salt-lime treatment on optimum moisture are shown for both soils in Figure 4.
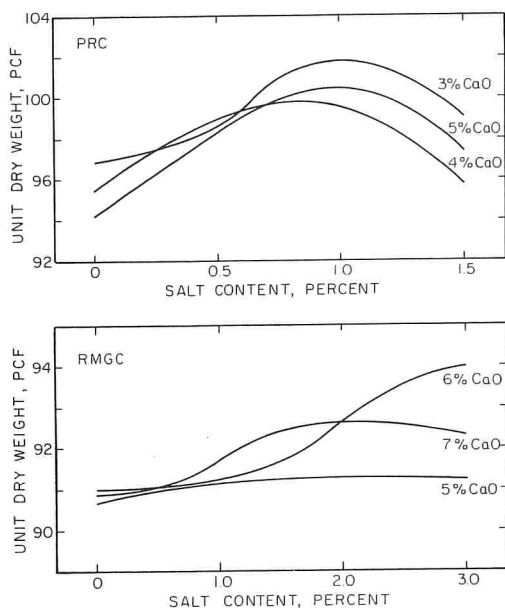

Figure 3. Effect of salt on compacted unit weight of PRC and RMGC mixtures near their lime modification optima.
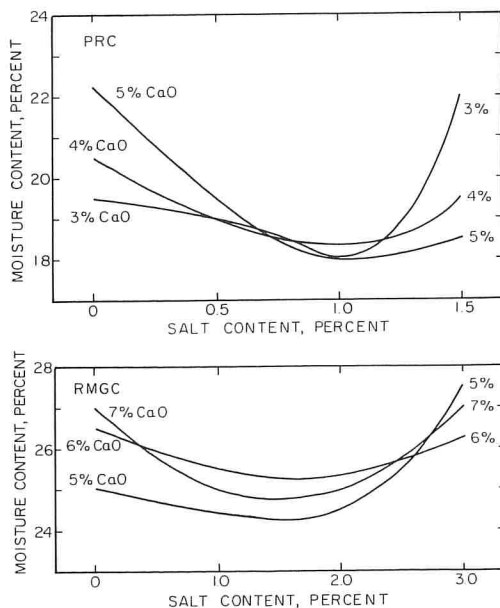
Figure 4. Effect of salt-lime treatment on optimum moisture contents of PRC and RMGC mixtures near lime modification optima.

It is thought that the small percentages required to cause these changes in unit weight and optimum moisture are sufficient to increase particle orientation by additional particle flocculation. Although this seems a sensible explanation, it is somewhat incomplete because flocculation should have occurred completely as a result of the addition of lime. Thus, compression of the double water layer must be enhanced by monovalent sodium ions, plus any dissolution effect caused by the presence of chlorine ions.

Workability of salt-lime mixtures was better than lime-treated mixtures. Added moisture was found to be more evenly distributed and constant after comparable curing times than was found with lime-treated soil. Ability of salt-lime admixtures to retain initial moisture contents was much greater than for mixtures of lime-treated soil.

Effects of Sodium Chloride-Lime Admixtures on Compressive Strength

Throughout this portion of the paper, results obtained from unconfined compression tests on salt-lime-treated soil will be compared with those obtained from lime-treated soil.

The loading rate for all unconfined compression tests was 0.03 in. per minute. This rate was based on a desired 5.0 percent strain in 10 minutes. Unconfined compression test specimens were molded in a Harvard miniature mold. Curing of all samples was accomplished by waxing specimens and placing them in a 25 C moist room for desired curing times. Each data point on all curves represents the average of at least 3 tests.

Initially, all strength tests were conducted on specimens compacted at optimum moisture content and maximum standard Proctor densities. Although differences between optimum moisture content and maximum dry density for the various mixtures will have some effect on strength, results obtained were expected to be more meaningful, for practical purposes, than samples prepared at the same compaction moisture content and unit dry weight.

Unconfined compression test results of samples compacted at optimum moisture contents and maximum standard Proctor densities indicate that the presence of salt increases the strength of both PRC and RMGC above that obtained from lime modification alone. Both lime-treated and salt-lime-treated PRC 7-day strengths passed through maxima as lime content was increased. RMGC mixtures showed a decreasing effect of salt on strength as lime content was increased. PRC showed greater response to salt-lime treatment at standard Proctor density than did RMGC. Results of 7-day unconfined compression tests are shown for both soils in Figure 5.

In order to determine the amount of strength gain contributed by salt, it was necessary to eliminate the variables of compacted moisture content and unit dry weight. Moisture-density curves for different compactive efforts were therefore obtained to determine a compaction moisture content and unit dry weight with corresponding compactive effort that could be used for all samples tested.

Samples thus prepared were cured in the manner described previously, with unconfined compression tests conducted after 7, 14, 21, and 28 days of curing. In all cases, admixtures of salt plus lime produced greater strengths than samples treated with lime alone. In PRC mixtures, 5 percent lime with 1 percent salt again showed a greater increase
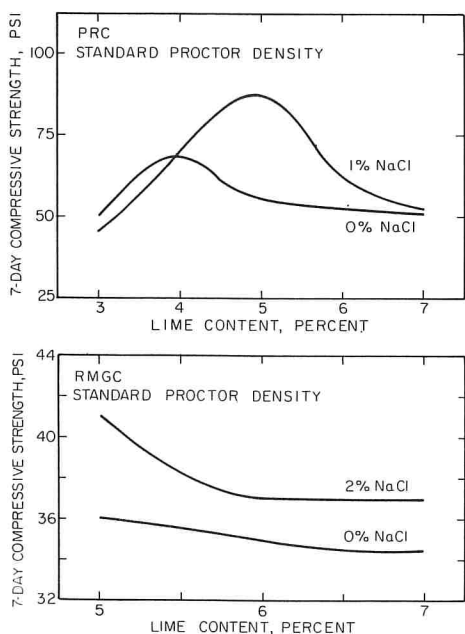

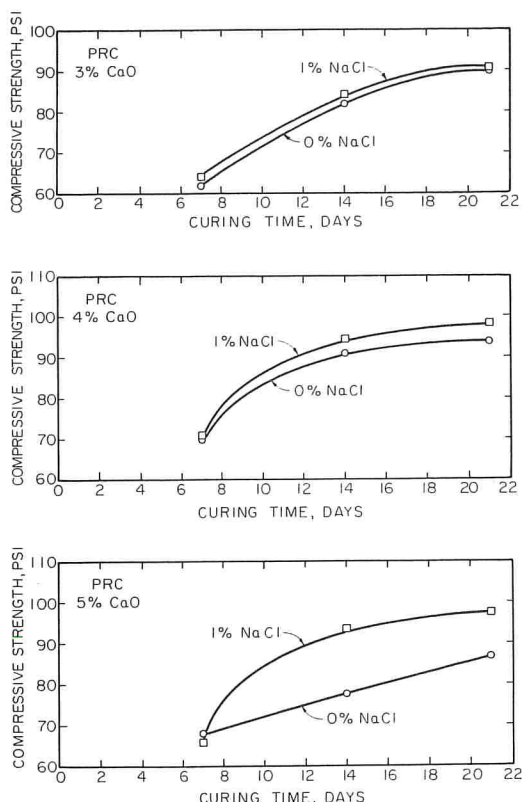Figure 5. Effect of salt on lime-treated PRC and RMGC strength when compacted to standard Proctor density.

Figure 6. Effect of curing time on strength of salt and salt-lime modified PRC samples under constant moisture and density conditions.
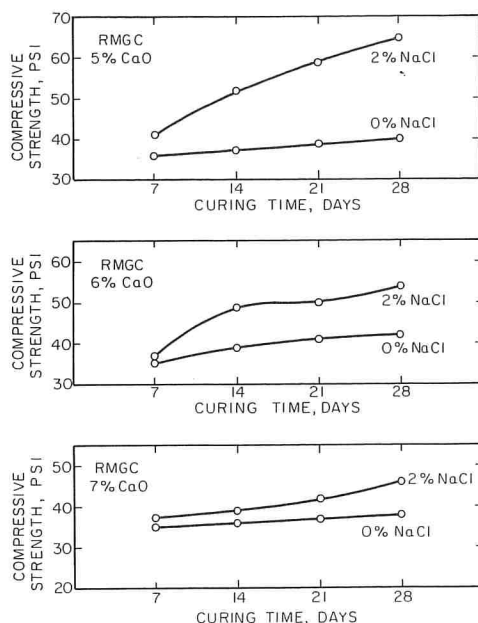

Figure 7. Effect of curing time on strength of salt and salt-lime modified RMGC samples under constant moisture and density conditions.

in strength than did either 3 or 4 percent lime with 1 percent salt. The same was true for RMGC. Mixtures of 5 percent lime plus 2 percent salt showed greater strength gain than did other percentages. The rate of strength gain was reduced as lime content increased. Figures 6 and 7 show strength gain relationships between lime-modified and salt-lime-treated PRC and RMGC respectively. Figure 8 shows the effect of lime content on compressive strength, with and without salt treatment.

Consistent gains in strength from salt-lime admixtures, above those obtained from lime-treated soil, indicate that salt may produce other effects besides flocculation in cohesive soil. Reaction of clay minerals with sodium chloride may create a situation where aluminum is more accessible to chemical reaction with lime. Because early strength of lime-treated soil results from formation of tetracalcium aluminate hydrates, sodium chloride may act as a catalyst in formation of these cementitious molecules. Increased solubility of silica or quartz in the presence of salt may well be the reaction that accelerates crystal growth, producing strength gain.

Discussion of salt-lime admixture strength thus far has been confined to lime percentages near the modification optimum for both soils. In this region of lime percentage, strength gain is usually of secondary consideration; however, it has been shown that strength gains obtained by addition of salt at modification lime contents are of fairly large magnitude. The next item to be discussed involves the benefit of adding small percentages of sodium chloride at lime contents around the stabilization optimum. At these lime contents strength is the primary, rather than secondary, concern.

In the case of PRC, a lime content of 8 percent was found to be stabilization optimum. Experiments were conducted with admixtures of lime and salt plus lime, 1 percent on either side and at optimum lime content. The greatest effect of salt on strength was
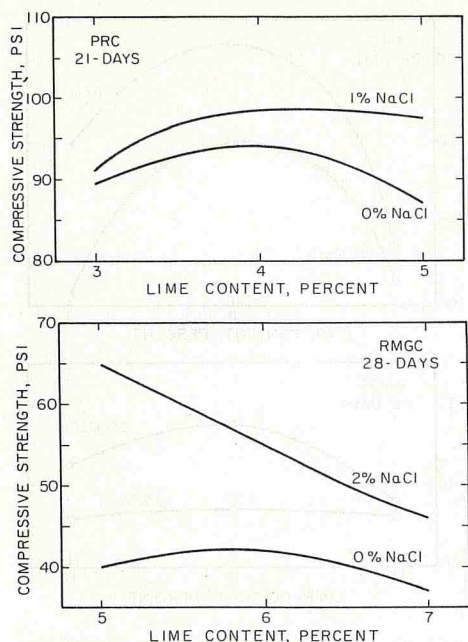
Figure 8. Comparison of lime and salt-lime compressive strength for PRC and RMGC near their lime modification optima under constant moisture and density conditions.
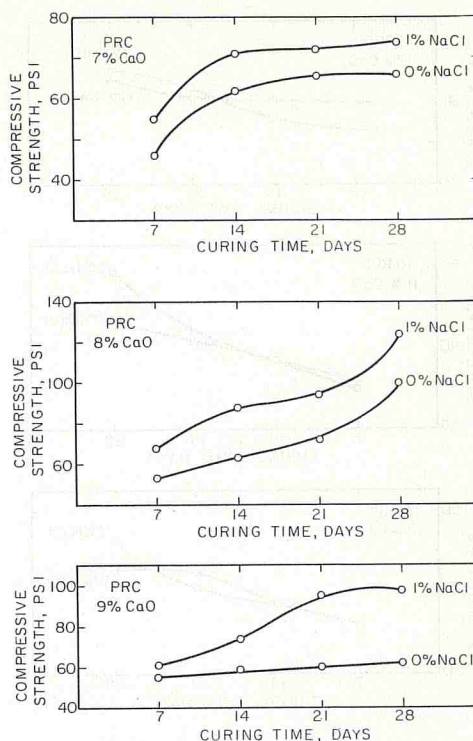

Figure 9. Effect of curing time on strength of lime and salt-lime stabilized PRC mixtures under constant moisture and density conditions.

found to occur at 8 percent lime content. The rate of strength increase was also found to be greatest at this lime content. Figure 9 shows the effect of curing time on strength for the 3 lime and 3 salt-lime mixtures tested. Addition of 1 percent salt at optimum lime content produced a compressive strength of 122 psi, which represents a 22 percent increase over lime alone.

RMGC was found to behave similarly to PRC, with maximum benefits obtained at a stabilization optimum of 11 percent lime. Although the rate of strength increase was substantial for RMGC mixtures, the total increase in strength was not as high as for PRC. The rate of strength increase is shown in Figure 10, which shows compressive strength as a function of curing time for all RMGC mixtures. The maximum strength, obtained at a lime content of 11 percent with 2 percent sodium chloride, was 49 psi, a strength increase of 11.5 percent over lime treatment alone. Thus, the effect of salt on strength gain is almost twice as much for PRC as for RMGC; however, because of the lower strengths obtained with RMGC, the latter increase may be of more importance. A summary of strength as a function of lime content, with and without salt, is shown for both soils in Figure 11.

The fact that the rate of strength gain was increased by addition of salt at all lime percentages would indicate that some type of catalytic reaction occurs during curing.

At this time, 2 purely hypothetical explanations are being considered. The first is that the sodium chloride reacts with clay minerals and upsets aluminum bonding in the clay, producing a condition where calcium ions may more easily unite with aluminum and silica to form new minerals, thus increasing the rate of strength gain. This reaction would have an effect on soil pH and charge equilibrium at the clay particle surface.
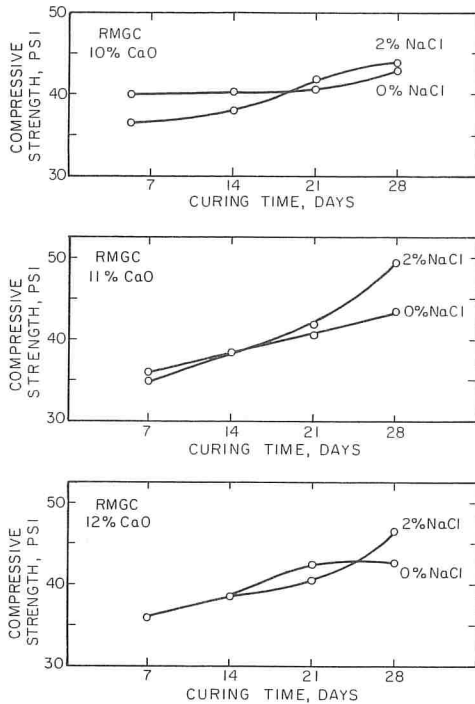
Figure 10. Effect of curing time on strength of lime and salt-lime stabilized RMGC mixtures under constant moisture and density conditions.
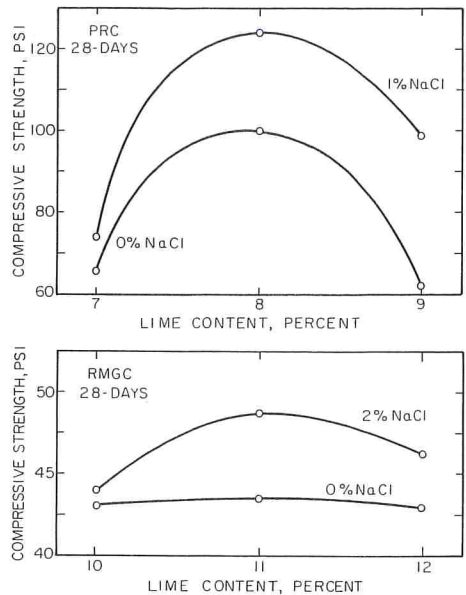

Figure 11. Effect of lime content on PRC and RMGC compressive strength without salt and at optimum salt content under constant moisture and density conditions.

The second explanation would be the increased solubility of silica in the presence of sodium chloride. By increasing the solubility, silica is available for reaction with calcium at a much greater rate than occurs in normal lime treatment.

SUMMARY

Based on data presented in this paper, the following preliminary conclusions are presented concerning effects of sodium chloride and sodium chloride-lime admixtures on the engineering behavior of cohesive soil. Results were obtained on 2 Oklahoma clay soils, but should be indicative of behavior for cohesive soils of similar origin and engineering properties.

1. Plasticity of cohesive soil is increased by addition of sodium chloride; however, PI increase at salt contents less than 2 percent is negligible.

2. Addition of sodium chloride was found to produce a substantial increase in compacted unit weight and corresponding decrease in optimum moisture content in the lower plasticity PRC, while the more plastic RMGC showed no response in compaction properties by addition of sodium chloride.

3. Both cohesive soils showed higher compacted unit dry weights and lower optimum moisture contents for salt-lime mixtures than for lime-treated soil.

4. Addition of small sodium chloride percentages with lime near modification optima did not increase plasticity sufficiently to change the material to nonselect subgrade soil.

5. Strength gains at both modification and stabilization optima were substantially increased by addition of small percentages of salt; thus sodium chloride increases the rate of strength gain and total strength associated with lime treatment.

6. Workability and moisture retention properties of both raw soil and lime-treated soil were enhanced by addition of small percentages of salt.

Sodium chloride may have use as a compaction aid, a moisture-retention agent, and a catalyst in increasing the strength of lime-modified and stabilized cohesive soils. Also, addition of small salt percentages counteracted the reduced unit dry weights and increased optimum compaction moisture contents associated with lime treatment of cohesive soils. Research currently in progress is expected to obtain more data concerning engineering behavior of salt and salt-lime-treated cohesive soils, and also to provide reasons for the behavior observed.

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Evaluation of Remolded Field Samples
of Lime-Cement-Fly Ash-Aggregate Mixtures

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• IN LATE SEASON CONSTRUCTION using lime-cement-fly ash-aggregate (LCFAA) mixtures, the reaction process may be greatly retarded by cool temperatures. There is some concern that as free water percolates through the pavements under these conditions there will be a tendency for the lime and cement to migrate and/or be leached from the mixture. If such is the case, and if a significant quantity of water percolates through the mixture, there may not be sufficient lime and cement available for proper reaction when the temperature rises the following spring.

Construction of the runways and taxiways at the Newark Airport provided an excellent opportunity to collect the necessary materials for a study to determine if lime and cement does migrate or is leached from the materials during adverse climatic conditions. Placement of the LCFAA material was started in late summer 1968 and continued through late November. During the latter portion of the construction season, a significant amount of rain fell on newly constructed pavements. Also, the mean daily temperature in the project area during the latter part of the 1968 construction season was in the low to mid 40's, which is sufficiently low so that little or no reaction would be expected to occur in these materials.

Construction procedures used in the Newark Airport were sufficiently controlled so that reasonable uniformity could be expected in the LCFAA mixtures. Layers of compacted LCFAA mixtures were placed in layers up to 30 in. thick and provided an excellent opportunity to study the migration of lime and cement throughout the depth of the pavements as the water percolated through the material. Also, the LCFAA materials used had sufficient permeability so that the water could percolate freely through the material thus providing ideal conditions for leaching to occur.

A laboratory investigation was designed to determine the extent of leaching or migration of the lime and cement under the field conditions described and the effect of the leaching and curing conditions on the subsequent curing of the LCFAA materials.

MATERIALS

Field samples of the LCFAA materials were taken from the pavement locations given in Table 1. Samples of the coarse aggregate, sand, and fly ash were obtained from stockpiles of materials used in the construction of these facilities. Characteristics of the coarse aggregate, sand, and fly ash are given in Table 2. Samples of lime were obtained from the suppliers and included both high calcium and monohydrated dolomitic limes. Portland cement used in the study was obtained from local supplies.

Field samples were taken from the airport pavements in 2-in. increments throughout the depth of the pavements. Thicknesses of the materials in place varied from 6 to 30 in. (Table 1).

When collecting the field samples, the material was placed in double plastic bags and carefully sealed to prevent moisture loss during shipment. The samples were shipped to the laboratory by commercial truck. The samples were shipped during early January and were frozen when received. They were stored in the frozen condition until preparation for testing to prevent further reaction of the constituents.

TABLE 1

LOCATIONS OF SAMPLES FROM NEWARK AIRPORT

Sample Location	Pavement	Station	Centerline Reference	Mix Designation	Depth (in.)
1	4L-22R	50+00	70 ft W	A	0 to 8
				B	8 to 24
				C	24 to 30
2	Taxiway B	19+00	On centerline	A	0 to 8
				B	8 to 24
				C	24 to 30
3	4L-22R	49+25	75 ft E	A	0 to 8
				B	8 to 16
				C	16 to 22
4	Taxiway A	10+00	10 ft E	B	0 to 8
5	Taxiway A	10+00	10 ft W	C	0 to 6
6	Taxiway A	38+00	5 ft W	C	0 to 6
7	Taxiway A	9+00	10 ft E	B	0 to 8
				C	8 to 14
8	Taxiway B	56+50	25 ft W	A	0 to 8
9	Taxiway A	37+00	10 ft W	C	0 to 6

Moisture content determinations were made on all samples as obtained from the field. A 600-gram sample was taken from each field sample for titration and pH tests to determine the lime and cement contents. The remainder of the material was set aside for strength and durability testing.

Mix formulas for the materials used in the pavement construction are given in Table 3. Optimum moisture contents and maximum dry densities for these mixtures are also given in Table 3. All mix proportions are given on a dry-weight basis.

TEST PROGRAM

The test program consisted of (a) titration and pH tests on all field samples (60); (b) strength and durability tests on selected samples of remolded material (30); and (c) a laboratory study to evaluate leaching under controlled conditions. Miscellaneous tests as necessary to develop the standards for comparison were also conducted.

TEST PROCEDURES

Titration Test

A titration procedure was used to determine the lime and cement content of the field samples. Details of the procedure are presented elsewhere (1). Essentially the procedure consists of dissolving the calcium (and magnesium) from the mixture in a solution of ammonium chloride and then determining the calcium concentration in the

TABLE 2

PARTICLE SIZE DISTRIBUTION OF COARSE AGGREGATES, SAND, AND FLY ASH USED IN THIS STUDY

Sieve Size	Percent Passing		
	Coarse Aggregate	Sand	Fly Ash
1 in.	100		
3/4 in.	41	100	
1/2 in.	5	100	
No. 4	—	98	100
No. 40		51	99
No. 80		13	—
No. 200		1	80
No. 325		—	64

Note: Loss on ignition of fly ash was 13 percent.

TABLE 3

FORMULATIONS AND COMPACTION CHARACTERISTICS OF MIXTURES A, B, AND C AS USED IN THE NEWARK AIRPORT

Item	Mix Designation		
	A	B	C
Mix proportions (dry weight basis)			
Coarse aggregate, percent	30.0	0	0
Sand, percent	52.5	83.0	83.5
Fly ash, percent	13.0	13.0	13.0
Lime, percent	3.6	3.2	2.8
Portland cement, percent	0.9	0.8	0.7
Optimum moisture content, percent	6.4	9.0	9.0
Maximum dry density, lb/cu ft	137.2	125.6	125.6

Note: For cold weather construction the cement is replaced with equivalent amounts of lime.

solution by titrating with a disodium salt of EDTA (ethylenediaminetetraacetic acid). The calcium concentration in solution is determined by comparing the EDTA necessary to titrate the solution from the field samples with a standard curve prepared from mixtures with known concentrations of lime and cement. Separate calibration curves are required for each mix formula and each lime type. Calibration curves for the materials used in this study are available in Appendix A.

pH Determination

Fifty grams of material from the field samples were charged into a plastic vial and approximately 100 ml of distilled water was added. After vigorous shaking for approximately 1 minute, the sample was allowed to settle for approximately 2 minutes and the pH of the fluid then read with a direct reading, electrode-type pH meter.

Strength Test

Unconfined compression tests were conducted on 2- by 4-in. cylindrical specimens made from remolded samples of material taken from the pavements. The specimens were cast in a solid steel mold and compacted in 3 approximately equal layers with a falling mass impact hammer with a 2-in. diameter face. The compacted specimens were pushed from the mold, cured in a sealed plastic bag at the prescribed temperature for the prescribed period of time, soaked, capped with hydrocal, and tested at a strain rate of 0.05 in. per minute.

Material for the cylindrical specimens was prepared by mixing for approximately 3 minutes in a Lancaster counter current mixer with a muller attachment. Water was added during mixing as required to bring the material to the desired (optimum) moisture content. The muller attachment effectively pulverized any chunks of material present in the field samples of the LCFAA material. Field samples with moisture contents above optimum were dried back to just below optimum prior to mixing. Laboratory prepared mixtures, which were used for comparison purposes, were blended and dry-mixed for 1 minute before adding water and 2 minutes after water was added.

Durability Test

The durability characteristics of the field material were evaluated by evaluating the potential of remolded field samples to develop and retain compressive strength after a short curing period and repeated freeze-thaw cycles.

Six 2- by 4-in. cylindrical specimens were made using the procedure outlined for the compressive strength test. After compaction these specimens were sealed in a plastic bag and cured for 2 days at 120 F. Three specimens were then removed and tested in compression, and the remaining 3 specimens were bedded in moist sand and placed in a freezer for 16 hours. After 16 hours in the freezer the specimens were removed and allowed to thaw for 8 hours at approximately 70 F. During the thawing period the sand in which the specimens were embedded was kept very moist, approaching saturation. After the prescribed number of freeze-thaw cycles, the embedded specimens were covered with plastic to prevent moisture loss, stored for 7 days at 120 F, then soaked for 4 hours, capped with hydrocal, and tested in compression.

Leaching Test

A test program was undertaken to evaluate leaching of the lime and cement from the LCFAA mixtures under laboratory conditions. Four lucite tubes, approximately 2 ft

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[1]The original manuscript of this paper included Appendix A, Calibration Curves Used to Determine the Lime Plus Cement Content of the LCFAA Mixtures, and Appendix B, Data From Tests on Field Samples of LCFAA Mixtures. The 2 appendixes are available in Xerox form at cost of reproduction and handling from the Highway Research Board. When ordering, refer to XS-29, Highway Research Record 315.

long and 4 in. in diameter, were filled to a depth of approximately 18 in. with a labora-
tory prepared mixture (Mix B). The mixture was brought to optimum moisture content,
and the proper quantity of material was charged into a lucite tube and vibrated to the
desired density. After compaction, specimens were covered and allowed to cure for
approximately 48 hours at a temperature of approximately 70 F. After 48 hours of cur-
ing 2 samples were treated with a prime coat (MC-0 applied at a rate of 0.25 gal/sq yd),
and all 4 specimens were placed in a controlled temperature cabinet and held at a tem-
perature of between 35 and 40 F.

Water in appropriate quantities to simulate the rain that fell on the pavements was
poured on each specimen and allowed to percolate through the LCFAA material. The
water was placed on the specimens in quantitative increments to simulate the amount of
rain that fell during specific periods on the pavements. For example, on November 10,
1968, approximately 1 in. of rain fell on the pavements between 1 a.m. and 4 p.m. To
simulate this condition sufficient water was placed on the laboratory samples at 7 a.m.
to give the equivalent of $\frac{1}{2}$ in. of rainfall; at 11 a.m. water equivalent to an additional $\frac{1}{4}$
in. of rain was added, and the same amount was added again at approximately 4 p.m.
It was observed that all water from previous applications had completely seeped away
prior to all subsequent applications. All water that percolated through the mixtures
was caught, retained, and tested for calcium content using the titration test.

When the amount of water equivalent to the rainfall for a 10-day period after place-
ment had percolated through the samples, the material was removed from the tubes and
subjected to the same test program as the field samples.

PRESENTATION AND DISCUSSION OF FINDINGS

Data from tests on field samples are given in detail in Tables B-1 through B-6 in
Appendix B.[2] Some of these data are reproduced in different format in the body of this
report to facilitate the discussion.

Leaching of Lime and Cement

It is expected that if lime and cement were leached from the LCFAA mixtures it
would produce several significant changes in the material. If leaching is fairly com-
plete it can be expected that the pH of the material will drop to below the 12.0 to 12.3
range (2). It is also expected that with significant leaching there would be a marked
reduction in the lime plus cement content as determined by the titration test. It also
seems reasonable to expect that, if sufficient calcium is leached from materials in the
upper layers to produce a calcium-saturated water percolating through the material,
some of this calcium will be deposited in the lower layers thus producing an increase
in the indicated lime plus cement content of the material from the lower depths.

A study of the pH data from the field samples shows that the pH of only 3 samples fell
below 12.0, and only one of these had a pH below 11.0. Generally, free lime still
is present in the material and available to react under favorable conditions.

Results from the titration tests on the field samples are shown in Figure 1. Two
curves are shown: The curve for the lower lime plus cement content is based on a 1-
minute stirring time in the NH_4Cl, and the curve for the higher lime plus cement con-
tent is based on a 5-minute stirring time. These 2 stirring times were used in lieu of
the standard 2-minute stirring time in an effort to distinguish between the lime and ce-
ment that was readily dissolved in the NH_4Cl, and that which required a longer dissolu-
tion time. As seen in Figure 1, the curves for the 1- and 5-minute stirring times are
similar in shape and, for practical purposes, are parallel.

Two features are noted in the data shown in Figure 1. First, the lime plus cement
contents determined by the titration tests are generally lower than the lime plus cement
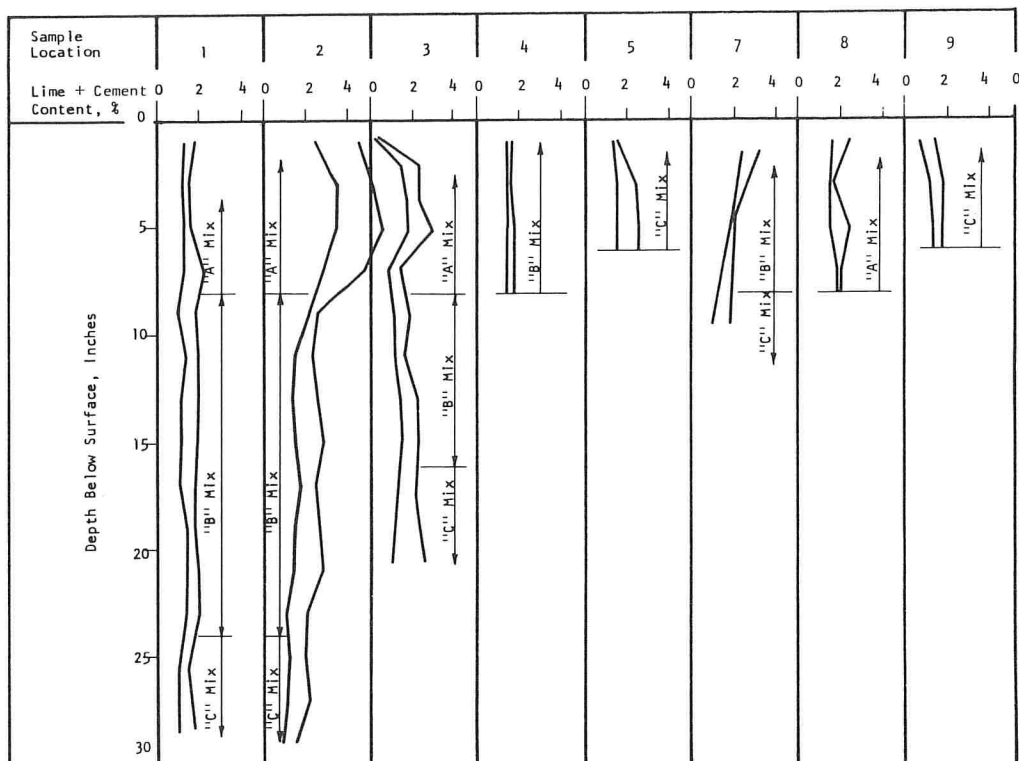
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[2] See footnote 1.

Figure 1. Lime plus cement content of the field samples as determined by the titration test.

content specified for these materials, and, second, the lime plus cement content determined with a 5-minute stirring period is consistently higher than that determined with the 1-minute stirring period. These data suggest that not all lime and cement are dissolved by the NH_4Cl, at least not during the relatively short mixing times used in this study. There is also a question of how much influence the curing time and temperature have on these results.

To develop further information on the effect of curing and stirring times on the indicated lime plus cement content, a series of specimens of mix B were made and cured for varying times in sealed bags at 120 F. These specimens were then pulverized in the same manner as the field samples and the lime plus cement contents determined using the titration test procedure. Results from this study are shown in Figure 2. In general, it is seen that as the curing time increases the lime plus cement content indicated by the titration procedure decreases. The reduction in the indicated lime plus cement content is especially rapid during the first 2 days of curing. Results shown in Figure 2 also indicate that as the curing time increases longer stirring times are needed to remove the comparable quantities of lime and cement from the mixture. The indicated lime plus cement content after 4 to 7 days of cure in the laboratory at 120 F is about the same as that obtained from the field samples.

Results from the leaching study on the material in the lucite tubes in the laboratory also indicate a relatively small amount of calcium is leached from the samples. The lime plus cement contents determined by the titration tests on the laboratory specimens are given in Table 4. The average lime plus cement contents of 1.95 percent with 1-minute stirring and 2.22 percent with 5-minute stirring are not significantly different from lime plus cement contents obtained from the field samples using identical test procedures.
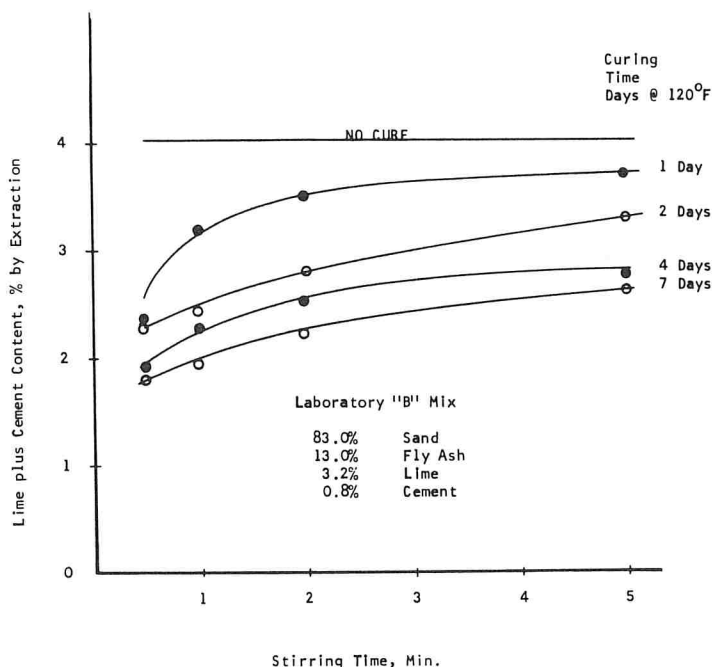
Figure 2. Effect of stirring time on lime extraction on samples with varying curing times.

Titration tests on the water that percolated through the LCFAA materials in the tubes indicated that less than 0.1 percent (of the approximately 4.0 percent added to the mixture) of lime plus cement had been dissolved in the water as it percolated through the mixture.

Strength and Durability Test Results

Durability as used in this presentation refers to the ability of the material to regain or retain compressive strength under repeated cycles of freezing and thawing and subsequent curing. Thus, strength and durability of these materials are closely related, and the test results are discussed together.

Figure 3 shows the age-strength relationship of LCFAA mixtures prepared in the laboratory. The 2 lower lines represent the data from the original mix design on these materials (3), and the upper line is from a mixture made and cured in the laboratory using the mix B formula. Numbers in the parentheses on the 2 lower lines denote the ratio of the strength of the material after it had cured for 1 day at 120 F and was then remolded and recompacted to that of the undisturbed material.

The effect of time between placement and sampling and subsequent remolding on the strength development of LCFAA field samples is shown in Figure 4. These data indicate that as the period between placement and sampling increased the strength developed in

TABLE 4

RESULTS FROM TITRATION TESTS ON LABORATORY LEACHING STUDY

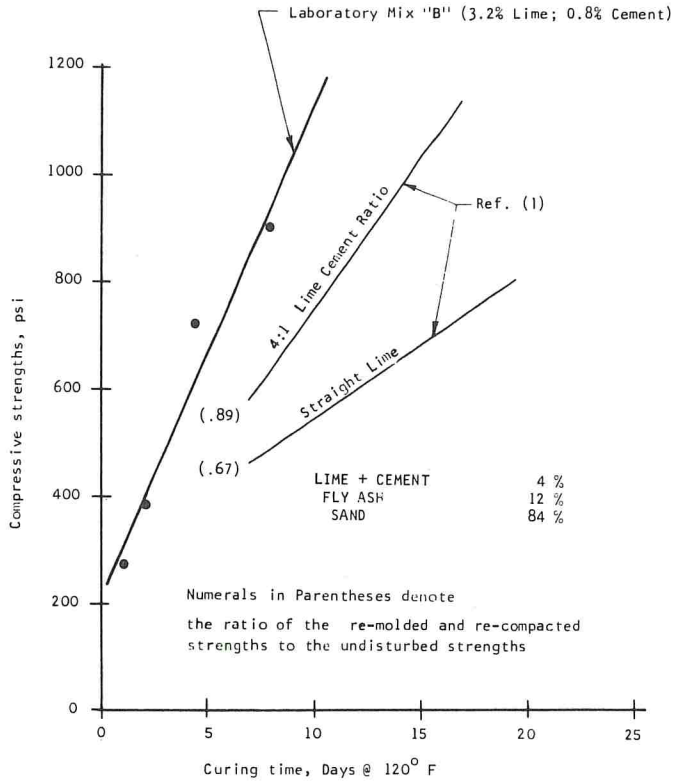| Tube	Percent Lime Plus Cement Content After Mixing in NH_4Cl	
	1 Minute	5 Minutes
1	1.9	2.0
2	2.1	2.5
3	2.0	2.0
4	1.8	2.4
Average	1.95	2.225

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Figure 3. Relationship between curing time and compressive strength and between remolded and undisturbed samples.
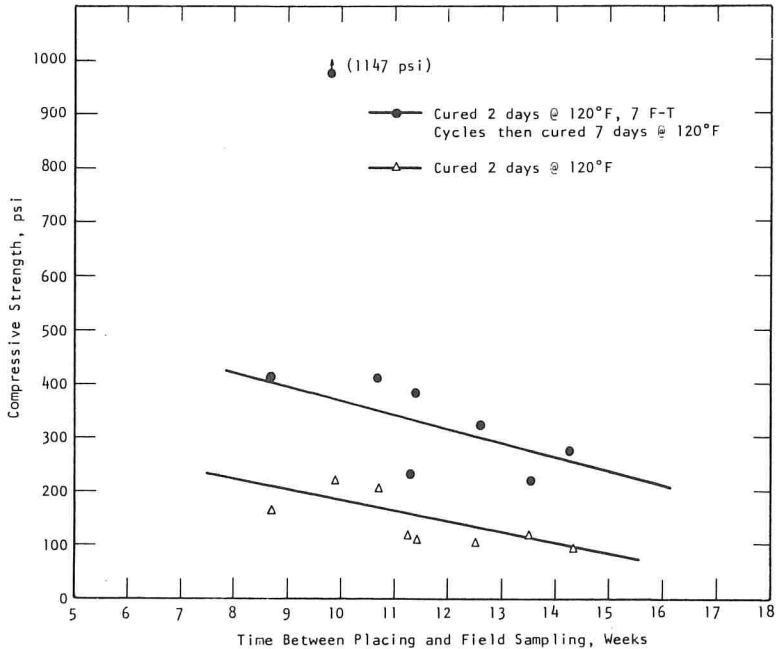

Figure 4. Effect of time between mixing and placing and remolding on the compressive strength of the LCFAA materials in the field.

the remolded material decreased. This is as expected, as the more lime and cement utilized in the reaction process before remolding the less there is available to react after remolding and recompaction. This should be kept in mind in evaluating the subsequent data on the strength development of remolded and recompacted materials from the field samples.

Figure 5 shows the effects of alternate freeze-thaw cycles and accelerated curing on the strength development of the laboratory prepared LCFAA mixture. When the material has had a relatively short curing time, alternate cycles of freezing and thawing may cause a significant reduction in the compressive strength. This strength is quickly recovered, however, when curing conditions are favorable as is shown in Figure 5 for the second 2-day curing period. After the second curing period, the material gained strength during the freeze-thaw cycles and had a further substantial gain in strength during the 2-day curing period following the second series of freeze-thaw cycles.

Figure 6 shows the strength gain and loss pattern for the undisturbed laboratory mix of LCFAA material plus a probable curve of the disturbed material developed from tests using materials from the field samples. The strength of the remolded material from the field samples after 7 freeze-thaw cycles was not determined as the upper $\frac{1}{2}$ to 1 in. of the specimens appeared soft so that effective capping was not possible. After an additional 7 days curing at 120 F, the softened materials rehardened sufficiently to permit the specimens to be capped and tested in compression.

The strength of the remolded field samples of LCFAA material after 2 days curing at 120 F was of the order of one-half the strength of freshly mixed material for the same curing conditions. There is no direct comparison that can be made of relative strengths of the undisturbed and remolded materials after the freezing and thawing, but examination of the trends of the data indicates that the remolded materials did develop a substantial proportion of their potential strength as indicated by the results from tests on

LABORATORY MIX

Proportion "B" Mix

83% Sand
13% Fly Ash
0.8% Cement
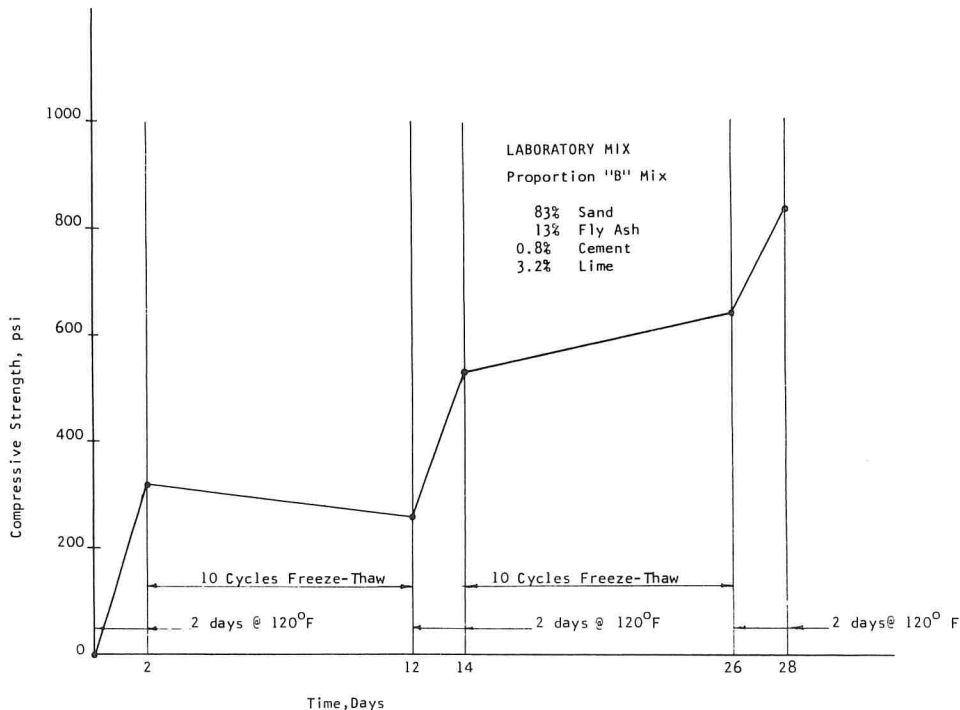3.2% Lime

10 Cycles Freeze-Thaw

2 days @ 120°F

Figure 5. Effects of freezing and thawing plus curing on compressive strengths of lime-fly ash mixtures.
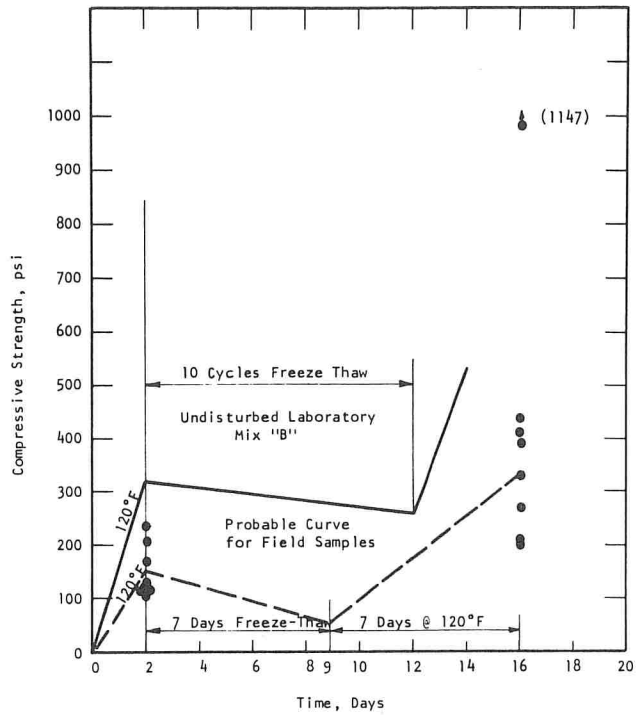
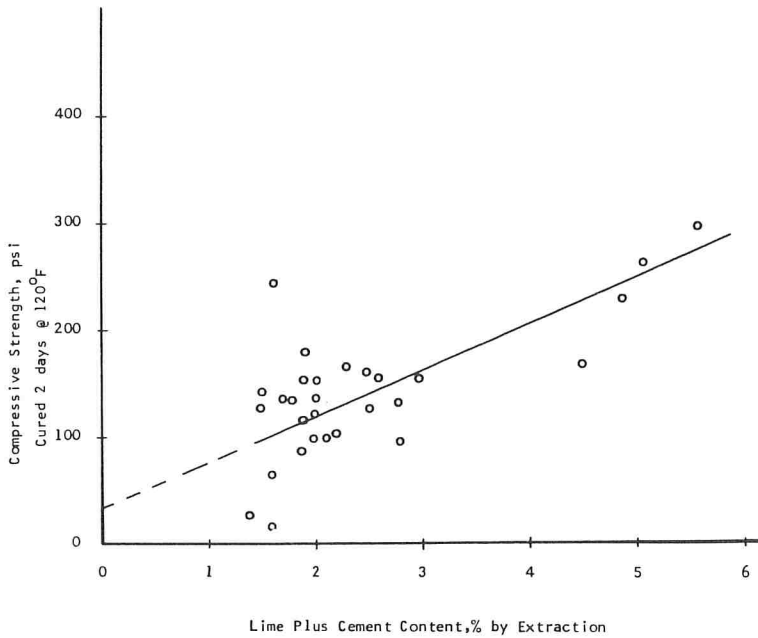Figure 6. Comparison of durability of disturbed and undisturbed samples.

Figure 7. Correlation between lime plus cement content and compressive strength of field samples.

the undisturbed material. Considering the effect of time between compaction and re-molding on the field samples, these results suggest that the materials in place should develop adequate strength and durability under favorable curing conditions during the spring and summer months.

Figure 7 shows the relationship between the lime plus cement content indicated by the titration test and the compressive strength developed in the remolded field samples. The natural scatter in the test results plus the limited number of test results available make it impossible to establish a precise correlation. There appears, however, to be a strong trend toward higher strengths with higher indicated lime plus cement contents.

CONCLUDING REMARKS

Findings from this program indicate that the lime and cement in the LCFAA mix-tures used in the Newark Airport pavements are stable and are not subject to signifi-cant leaching or migration over short periods of time. More specifically, the findings indicate that those LCFAA materials placed late in the construction season will retain nearly all of the lime and cement that were added during mixing and that this lime and cement will react with other components of the mixture under favorable conditions even after being dormant for a considerable period.

Findings from this study indicate that the more extensive the curing is before freez-ing and thawing the less damage will be done to the materials by cyclic freezing and thawing. Also, materials damaged by freezing and thawing will recover a significant portion of the lost strength under favorable curing conditions provided sufficient lime and cement are retained in the mixture to support continuing reactions. It was observed in this study that, when the LCFAA materials are subjected to alternate freeze-thaw cycles under the conditions used in this study, the most severe damage to the materials occurs near the top of the specimens for a depth of from 1 to 2 in.

Findings from this study seem to indicate that LCFAA materials can be placed later in the construction season than previously thought, provided proper precautions are taken. For example, it may be necessary that for late season construction the lime or lime and cement contents be increased to offset the effects of any initial set and damage caused by freezing and thawing to the ultimate strength developed by the LCFAA mixture. Also, provision should be made to remove any loosened or damaged materials from the surface prior to placing subsequent layers during spring and summer.

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Differential Thermal and Thermogravimetric Analysis of Reacted Bentonite-Lime-Water Mixtures

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Thermal methods are applied to supplement previous results by X-ray diffraction of mixtures of bentonite with calcitic, monohydrated, and dihydrated dolomitic lime reacted in the presence of water at 23 C for 3 years in sealed containers. In addition, the cured mixtures were reacted at approximately 100 percent relative humidity at temperatures of 40 and 80 C for 4 to 5 days. These mixtures were then subjected to hydrothermal treatment at several intermediate temperatures to 170 C. X-ray diffraction indicated calcium silicate hydrate (CSH) gel, CSH I, and possibly CSH II in all room-cured mixtures, with C_4AH_{13} indicated only in calcitic and monohydrated lime mixtures. Better crystallized CSH phases, including tobermorite, resulted from hydrothermal treatment. In most instances, results by X-ray diffraction were confirmed using thermal methods. In addition there were new findings; it was possible to characterize reaction products having low degrees of crystallinity. A chlorite-like mineral was noted in all mixtures containing dolomitic lime. CSH II was also clearly identified as a reaction product in most of the mixtures, particularly those reacted at intermediate temperatures.

•A COMPLETE EVALUATION of a stabilized soil involves, among other factors, identification of products of the reaction between the additives and components in the soil. This is a difficult process, particularly when X-ray diffraction is the only analysis technique employed and the reaction products have low degrees of crystallinity. This is often the case, particularly in soils stabilized with hydrated lime under field temperature conditions. One supplementary technique is Differential Thermal Analysis (DTA), accompanied sometimes by Differential Thermogravimetric (DTG) and Thermogravimetric (TG) analysis to obtain weight loss and other reaction data.

The present study applies these thermal techniques to the analysis of separate reacted mixtures of a pure clay mineral with 3 types of lime in water.

Previous studies by X-ray diffraction of these mixtures reacted at 23 C using calcitic and monohydrated dolomitic lime indicated formation of the tetracalcium aluminate hydrate (C_4AH_{11-12}). These mixtures as well as the one reacted at 23 C using dihydrated dolomitic lime showed that calcium silicate hydrate (CSH) gel, $CaCO_3$, CSH I, and possibly CSH II were also produced after curing for long periods, with apparently increasing degrees of crystallinity with longer reaction time. This phenomenon in the hydrothermally reacted mixtures was evident, with well-crystallized tobermorite appearing at the highest temperatures and with other CSH phases at intermediate temperatures. In all mixtures the products noted at 23 C were found to persist beyond the hydrothermal reaction at 105 C (1). Reexamination of the X-ray diffraction patterns of dolomitic monohydrated lime mixtures revealed that a 15.2 Å nonexpanding mineral was produced in hydrothermal reactions at 105 C and above.

For obvious reasons, previous DTA, DTG, and TG studies of the 2 systems, CaO-SiO₂-H₂O and CaO-Al₂O₃-H₂O, were selected from the literature and used in the present analysis.

DTA of the compound C_4AH_{12-13} by Turriziani showed weak endotherms near 130 and 170 C with a strong endotherm near 230 C. Another weak endothermic peak near 330 C was followed by a broad weak exothermic bulge near 350 C (2). Carlson's studies of the calcium aluminoferrite hydrates explained the progressive dehydration of C_4AH_{13} to be as follows: The 11 hydrate is formed at 127 C; 4 additional molecules of water are lost at 194 C. Above 250 C the hexagonal hydrate goes to C_3AH_6, the isometric form, accompanied by formation of $Ca(OH)_2$. C_3AH_6 is decomposed near 350 C (3). TG studies of cement chemistry by Longuet essentially confirm these findings (4).

DTA of CSH gel prepared at room temperature by Brunauer and Greenberg shows only a strong endotherm near 200 C (5). Diamond noted this phenomenon, but his preparations also gave a small exotherm between 840 and 865 C (6). Kalousek found that CSH I having CaO/SiO_2 (C/S) ratios between 0.8 and 1.0 gave similar DTA patterns for both room temperature preparations and those hydrothermally reacted at 175 C. The peak temperatures increased with increases in C/S ratio but the exotherms were about 7 C lower for the room temperature preparations. After 4 days of autoclaving at 175 C, a 0.8 C/S mixture made with lime and silicic acid showed weak DTA effects, namely, an endotherm near 270 C and an exotherm near 840 C. At less reaction time, the low temperature endotherm moved from just below 200 C to slightly higher temperatures; this was accompanied by a progressive diminution in intensity of the 840 C exotherm. Kalousek indicated that an intermediate lime-rich phase (C/S = 1.75) interacted with unreacted quartz after about 6 hours (7, 8, 9). Gaze and Robertson hydrothermally reacted lime with quartz having a C/S ratio of 0.33 at 165 C to give a product showing a weak DTA endotherm at 570 C followed by a strong exotherm at 818 C (10). Diamond's hydrothermal preparations of tobermorite gave a noticeable endotherm near 275 C and a barely noticeable exotherm near 820 C. Aluminum substituted tobermorite produced the following changes in his DTA patterns: for 3 percent Al, the endotherm appeared at 810 C; for 5 percent Al, both endotherm and exotherm were 10 C lower than for pure tobermorite. Changes noted in the 10 percent Al pattern, as compared with that for 5 percent Al, included the appearance of a 630 C weak exothermic bulge and an 825 C exotherm; the 15 percent Al preparation showed 285 and 380 C medium endotherms, a 510 C weak exotherm, and an 835 C very strong exotherm (6).

Mackenzie reports that diluted 20 to 25 percent $CaCO_3$ mixtures show DTA peak temperatures between 830 and 940 C (11). Gaze and Robertson attribute a large endothermic effect at 780 C to calcite. This lower temperature is affirmed to be due to appreciable quantities of CO_2. A similar result was noted by Mackenzie in DTA findings for a calcium carbonate sample from carbonation of $Ca(OH)_2$. Lowering of the calcite peak temperature was attributed to the finer particle size of the secondary carbonate. Longuet reports a weight loss study in which decomposition of $CaCO_3$ in air commences at 630 C and terminates at 880 C (4, 10, 11).

Van Bemst presents DTA and DTG studies of CSH I (C/S = 1.36) and CSH II (C/S = 2.01). Exothermic effects near 155 and 800 C appeared on DTA patterns for both preparations. CSH I showed a very strong exotherm at 840 C while CSH II showed twin thermal effects between 340 and 380 C as well as weak exotherms at 850 and 920 C. The greatest rate of weight loss occurred below 200 C, as indicated by the DTG curve. Weak decomposition reactions also occurred near 400 C with intermediate reaction rates near 710 C for both compounds. The distinguishing features noted on the DTG patterns were the intermediate reaction at 260 C for CSH II and the one at 640 C for CSH I (12). Unidentified calcium aluminate hydrates and calcium silicate hydrates were found by X-ray diffraction in various lime-clay mixtures as early as 1960 (13, 14). DTA, as well as X-ray diffraction, was used in the analysis of lime reacted with several clay minerals to characterize reaction products, namely, C_4AH_{13}, 10 Å tobermorite, and monocarbonate aluminate (15).

Among the results of X-ray diffraction studies of products of lime-bentonite-water mixtures by McCaleb were the following: With $Ca(OH)_2$, room temperature mixtures produced calcite and Ca-montmorillonite. With $Mg(OH)_2$, Mg-montmorillonite, brucite,

and a small amount of vermiculite-chlorite-like mineral were formed. When both $Ca(OH)_2$ and $Mg(OH)_2$ were present, no such changes were noted. Hydrothermally reacted (168 C) mixtures contained a 12.8 Å and 15.7 Å montmorillonite as well as tobermorite and calcite. Little difference was noted between room temperature and hydrothermal preparations using $Mg(OH)_2$. When both $Ca(OH)_2$ and $Mg(OH)_2$ were used, a 12.9 Å montmorillonite, a 14.7 Å vermiculite, and an 11.7 Å tobermorite were formed (16).

Caillere et al. treated montmorillonite with $MgCl_2$ and NH_4OH solutions and described the product as having characteristics between chlorite and vermiculite. The explanation of the phenomenon was that $Mg(OH)_2$ rapidly precipitated between the structural layers of the montmorillonite. DTA results from artificially prepared chlorites showed a weak endotherm between 500 and 600 C and a weak exotherm near 850 C (17, 18, 19). Artificial pseudochlorites are reported by Mackenzie to show about 3 times the weight loss as that of a natural chlorite and that half occurs at about 300 C from interlayer water and a third near 400 C, attributed to brucite layer dehydration; the balance of the dehydration occurs slowly up to 525 C, at which temperature the montmorillonite layer decomposes (11).

Using X-ray diffraction, supplemented by DTA, Wang and Handy noted that MgO reacted at 23 C for 28 days with bentonite and water to produce a 15.2 Å nonexpanding material that was characterized as a chlorite-like mineral. The DTA peaks for this mixture reacted at 126 C for 1 day were as follows: weak endotherms at 540 and 810 C and a medium exotherm at 845 C. Curing for longer periods at 126 C resulted in an increased temperature and intensity for the low temperature endotherm and an increased intensity for the exotherm at a slightly lower temperature. After 8 months, the well-crystallized mineral serpentine was the final product, exhibiting longest X-ray diffraction spacing at 7.19 Å and DTA peaks as follows: a strong endotherm at 620 C and a very strong exotherm at 800 C. As the reaction proceeded from 1 day to 8 months, there was a progressive diminution of the 110 C hygroscopic moisture endotherm characteristic of montmorillonite (20).

McConnell performed a dehydration study of the natural 11.3 Å hydrate, tobermorite, from which he found that almost two-thirds of the weight loss occurred between 200 and 300 C. In the DTA of the same material, a strong endotherm was observed at 250 C. The 14.6 Å hydrate phase was also present in small quantity, which accounted for other dehydration effects at 105 C (21).

CSH phases with low degrees of crystallinity have always been difficult to identify by X-ray diffraction alone (1). The relationships between phase and degree of crystallinity of the various products are given in Table 1.

PROCEDURES

The clay mineral used was Otay, California, bentonite API reference clay mineral No. 24 (22). Lime, synthesized from reagent grade laboratory chemicals, was mixed with bentonite in water in the proportions given in Table 2.

Mixtures prepared in slurries were sealed in plastic containers to prevent loss of water or entry of carbon dioxide and cured at 23 C, ±2 deg, for 3 years. Reactions at 40 C for 5 days and 80 C for 4 days, as well as hydrothermal reactions of the cured

TABLE 1
RELATIONSHIP BETWEEN PHASE AND DEGREE OF CRYSTALLINITY

Product C/S Ratio	Degree of Crystallinity		
	High	Low	Very Low
0.8 to 1.33	Tobermorite: 9.3, 11.3, and 14 Å Well-crystallized CSH I	CSH I	Plombierite (natural gel)
1.5 to 2.0	Tobermorite: 10 and 12.6 Å	CSH II	CSH gel
X-ray diffraction pattern	Complete, including h, k, l reflections	Poor, with basal and hk or hko reflections	Very poor, with one or more hk reflections and no basal reflections

mixtures at 105, 145, 156, and 170 C for 12 hours, were employed. The hydrothermal procedure involved reaction at the vapor pressure for steam in an apparatus manufactured by the Parr Instrument Company, Moline, Illinois.

Samples from which specimens were taken were allowed to equilibrate under vacuum over $CaCl_2$ for 24 hours; they were then ground to approximately 200 mesh size before mounting on the DTA apparatus.

All reacted mixtures were analyzed using a DTA apparatus equipped with an automatic temperature controller, pro-

TABLE 2

LIME-BENTONITE-WATER MIXTURE

Weight Ratio	Starting C/S Ratio of Mixture
A. $Ca(OH)_2$:bentonite:H_2O 0.45:1.00:1.62	0.69
B. $Ca(OH)_2$:MgO:bentonite:H_2O 0.45:1.00:1.62	0.45
C. $Ca(OH)_2$+$Mg(OH)_2$:bentonite:H_2O 0.45:1.00:1.62	0.39

The bentonite formula is

$Al_{1.43}Fe_{.03}Mg_{0.64}(Al_{0.01}Si_{3.99})O_{10}(OH)_2(Na_{0.12} \frac{Ca}{2} 0.07)$.

viding for a heating rate of 10 C per minute to 1,000 C. A vertical furnace arrangement is used. Two vertical $\frac{3}{8}$-in. diameter by $\frac{1}{2}$ in. deep sample holes were symmetrically located in a stainless steel block with centers 1 in. apart. Number 22 platinum-platinum 10 percent rhodium differential thermocouples were used. The furnace temperature couple was a separate chromel-alumel junction inserted in a $\frac{3}{8}$-in. diameter by $\frac{1}{2}$ in. deep hole drilled into the bottom of the block. An inert sample of powdered alumina was placed in one hole with the sample in the remaining hole. Materials were tamped with a tightness from which reproducible results were obtainable.

In order to clarify certain of the thermal effects noted on the initial DTA curves, selected specimens from the desiccated samples that had been stored in capped bottles for periods exceeding a year were reexamined. DTG and TG curves as well as a second set of DTA curves were simultaneously obtained for mixtures using all 3 types of lime reacted at 23 C and for all hydrothermally reacted mixtures containing only the dolomitic monohydrated lime. The second apparatus, namely, the Mettler Thermoanalyzer, was used. It was operated at the same heating rate. This apparatus is designated as equipment A in Figures 3 and 4 for these mixtures whereas the other apparatus is designated as equipment B.

RESULTS

Results of the DTA for Otay bentonite shown at the top of Figures 1, 2, and 4 include an interlayer water endotherm near 150 C. Two other medium endotherms near 650 and 855 C are from expulsion of hydroxyl water. The sharp exotherm near 1,000 C is associated with the appearance of the second endothermic peak but its exact origin is unknown (11).

Mixtures Reacted at 23 C for 3 Years

Differential Thermal Effects Below 300 C for All Types of Lime—DTA curves for the mixtures using calcitic and dihydrated dolomitic lime are shown in Figures 1 and 2 respectively. DTG, TG, and DTA curves for the mixture using monohydrated dolomitic lime are shown in Figure 3; TG data for the other 2 mixtures are summarized in the text. The first reaction appears near 140 C and represents weight losses between 6 and $7\frac{1}{2}$ percent, representing expulsion of hygroscopic and interlayer water. Heat treatment of an identical sample in a 105 C oven for 24 hours resulted in a hygroscopic water loss of less than 3 percent; there was a 2 percent weight loss at the corresponding temperature on the TG curve, lower because of the relatively short period of heat application in the DTA apparatus. The total weight loss for the reaction near 140 C was low apparently because of additional desiccation during storage of the samples. Mixture constituents contributing to this initial thermal effect are unreacted bentonite, C_4AH_{13}, CSH gel, and CSH I, all of which were noted from previous X-ray diffraction study; thermal effects at higher temperatures that support these findings are noted later (1). Additional effects from C_4AH_{13} were detected in the mixtures using calcitic and monohydrated dolomitic lime. Dehydration of the hexagonal hydrate overlaps that
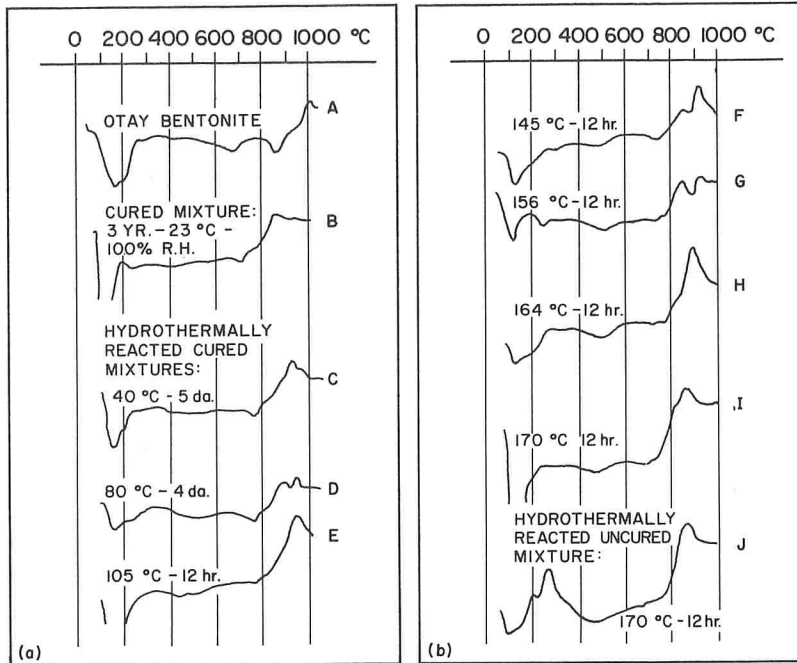
Figure 1. DTA curves of reacted mixtures of bentonite + Ca (OH)$_2$ and H$_2$O.

for the constituents mentioned earlier near 100 and 200 C (<u>2</u>, <u>3</u>, <u>4</u>, <u>6</u>). The endotherm centered near 250 C appears in all mixtures but stronger in the calcitic and monohydrated lime mixtures. This thermal effect may be associated with CSH I and II as well
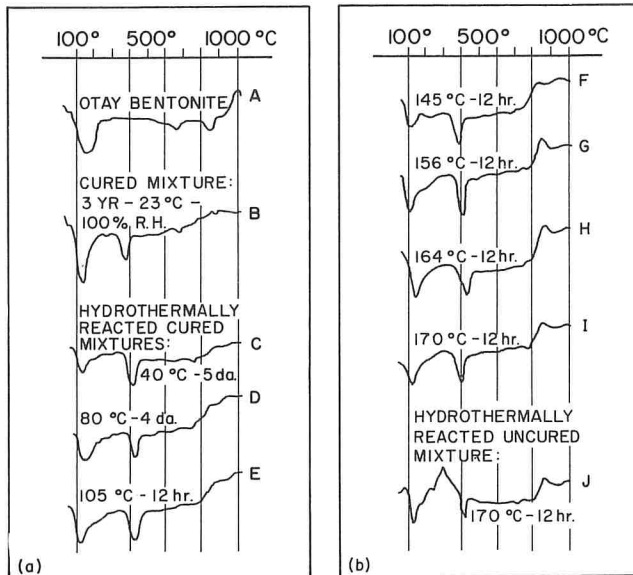

Figure 2. DTA curves of reacted mixtures of bentonite and Ca (OH)$_2$ + Mg (OH)$_2$ + H$_2$O.

as C_4AH_{13}; the aluminate hydrate has its most prominent DTA peak at 230 C, corresponding closely with TG data. Weight losses at this temperature for the calcitic and monohydrated lime mixtures were 50 to 100 percent greater than for the dihydrated lime mixture that contained no C_4AH_{13} ([1], [2], [9], [12]).

Differential Thermal Effects Above 300 C for Calcitic Lime Mixtures—Shown on DTA curve B in Figure 1 for this mixture reacted at 23 C, there are broad, diffuse, weak endotherms near 400 and 700 C, with weak undulating effects just above 500 C. These are followed by more prominent endothermic effects near 800 C and a medium exotherm at 840 C.

Between 300 and 500 C, there was a total weight loss of $4\frac{1}{2}$ percent. There were no significant peaks in the DTG curve in this temperature range although the weight loss curve reflected an increased rate commencing near 400 C and terminating near 560 C. The $1\frac{1}{2}$ percent weight loss between 300 and 400 C may be associated with dehydration of the C_3AH_6 phase that normally terminates near 350 C. $Ca(OH)_2$ dehydration in air usually begins near 400 C and terminates in the vicinity of 500 C and is associated with the remaining 3 percent weight loss ([3], [4]). An overlapping endotherm at 380 C and weak exotherm at 510 C from CSH II may contribute ([9]).

Beginning near 600 C, the DTG rate increases gradually to a diffuse peak near 700 C; the DTG reaction rate is equal to that noted at 140 C and represents a total weight loss of over 7 percent. These effects are attributed to the loss of hydroxyl water from unreacted bentonite ([11]). This is followed by a distinct DTG peak at 815 C at double the rate of weight loss of the preceding peaks, amounting to a loss of over 5 percent of the sample weight. This weight loss essentially terminates at 840 C, coinciding with the only distinguishable exotherm in the DTA pattern. The 815 C endotherm and accompanying weight loss is attributed to $CaCO_3$ ([4], [10], [11]). CSH I and II have strong weight loss reactions near 700 C and are also contributory to this thermal effect. CSH I usually shows a stronger exotherm near 850 C than CSH II, which has a second very weak exotherm near 920 C. CSH II also has a high rate of weight loss near 260 C that aids in distinguishing it from CSH I ([12]). Also contributing slightly to the 840 C exotherm is CSH gel that earlier was noted by a low temperature endotherm as well ([6]).

Differential Thermal Effects Above 300 C for Dihydrated Dolomitic Lime Mixtures—Shown on DTA curve B in Figure 2 for this mixture reacted at 23 C, there is a strong endotherm near 400 C with an associated weight loss of 4 percent, and several weaker, broad, and somewhat diffuse peaks appear at higher temperature. Most prominent are the weak endotherm near 560 C and the medium endotherm near 700 C. A weak endotherm at 730 and, appearing as a shoulder at 810 C, another endothermic effect preceded medium double exotherms at 840 and 920 C.

There are 5 essential differences in the thermal effects noted for this mixture when compared with those for the calcitic lime mixture. The 415 C endotherm represents decomposition of $Mg(OH)_2$. Second, thermal effects for the calcium aluminate hydrates are absent. In the third place, the appearance of a definite DTG peak at 560 C is accompanied by approximately twice as much loss of weight, namely, 3 percent. The fourth difference is associated with the endothermic effects near 700 and 800 C, where the weight loss measurement for the dihydrated lime mixture is 40 percent lower than that obtained for the other mixture. No clear indication for $Ca(OH)_2$ can be distinguished although noted by X-ray diffraction ([1]). The fifth difference lies in the diminished 840 C exothermic effect.

With regard to the third and fourth items just cited, the peak near 560 C is probably due to the interaction of the $Mg(OH)_2$ with the bentonite to produce a chlorite-like mineral having a low degree of crystallinity. Wang and Handy found that curing of a similar mixture at 23 C for a year produced a reacted mixture that gave a weak hump at 7 Å on the X-ray diffraction pattern. These findings were similar to those of McCaleb. However, X-ray diffraction of the Wang and Handy mixture, as well as that of the present mixture, indicated presence of expansive clay rather than the usual nonexpanding 15.2 Å peak ([1], [16], [18], [19], [20]).

Diminished intensity of the exotherm at 840 C and the accompanying 920 C exotherm as well as distinctive endotherms below 300 C and another endotherm at 710 C provide
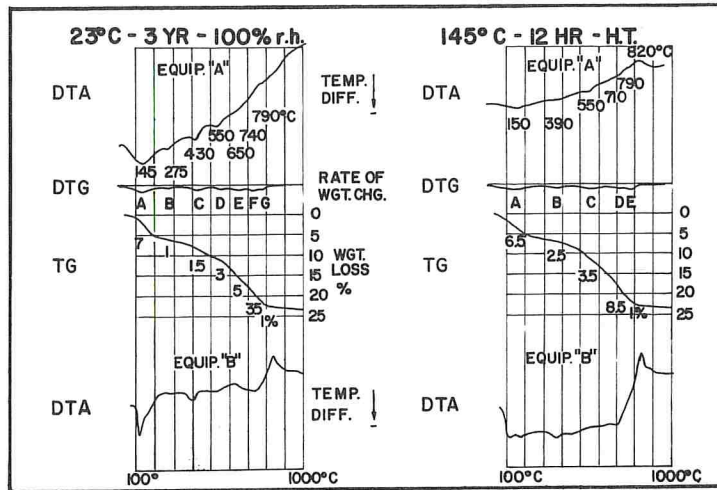
Figure 3. DTG, TG, and DTA curves of monohydrated dolomitic lime-bentonite-water mixtures.

the essential evidence for identification of CSH II in the mixture. It appears that contribution by CSH I to the 840 C exotherm is likely only if CSH I were present in small quantity and at a high degree of crystallinity (1, 7, 12).

Finally, the thermal effect near 700 C may be associated with dehydroxylation of unreacted bentonite as well as the aforementioned decomposition of CSH II (11, 12). The relatively lower weight loss for the dihydrated lime mixture at this temperature may be due to a diminished quantity of unreacted bentonite, due to its alteration to the chlorite-like mineral (16, 20).

Differential Thermal Effects Above 300 C for Monohydrated Dolomitic Lime Mixtures—On the left in Figure 3, the DTA for this mixture reacted at 23 C is shown. A medium endotherm at 430 C, with an associated DTG peak and weight loss of 1½ percent, is attributed to the decomposition of $Mg(OH)_2$. This effect is followed by a sim-
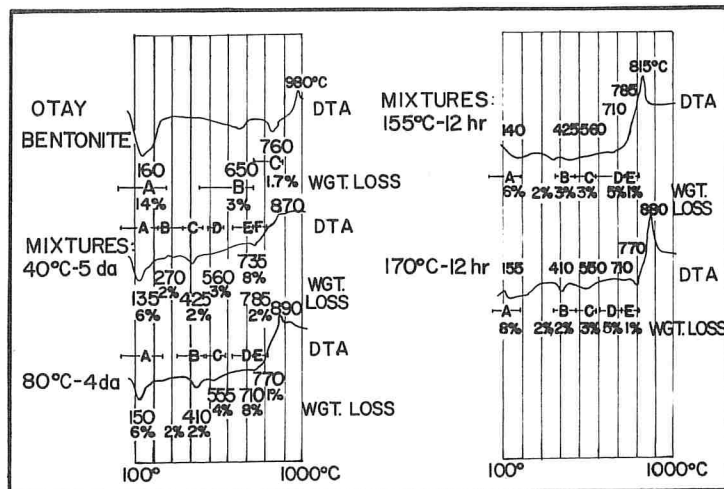
Figure 4. DTA curves of monohydrated dolomitic lime-bentonite-water mixtures.

ilar set of DTA and DTG peaks near 550 C, with a weight loss of 3 percent, which are associated with a chlorite-like mineral. Near 650 C the bentonite dehydroxylation peak appears on both DTA and DTG curves, accounting for a weight loss of 5 percent. Two DTG peaks appear to be combined into a medium endothermic effect between 700 and 800 C. The overall character of the 200 C endothermic effect and the associated exotherm at 840 C, noted particularly in the DTA curve by equipment B, is indicative of CSH I, to which the weight loss of $1\frac{1}{2}$ percent near 700 C is attributed. The breadth of the 700 to 800 C peak is probably associated with the 1 percent weight loss near 800 C. No distinguishable DTA effects are noted above 300 C for the final decomposition of the isometric phase of the initial C_4AH_{13}.

Hydrothermally Reacted Mixtures

40 C for 5 Days—DTA curves shown in Figures 1, 2, and 4 for all 3 mixtures differ most from those of mixtures cured at 23 C for 3 years by the diminished low temperature endothermic effects. Although evidence persists for the same constituents noted in the earlier discussion, the intensities are lower (2, 3, 6, 9). The second most striking change in the overall appearance of all 3 DTA curves is enhancement of the evidence for CSH II (9, 12). Effects may be noted for unreacted bentonite as well as $CaCO_3$ (4, 11, 12). $Mg(OH)_2$ decomposition may also be noted in Figures 2 and 4.

80 C for 4 Days—DTA curves shown in Figures 1, 2, and 4 appear about the same as for the 40 C reacted mixtures with respect to CSH gel, C_4AH_{13}, and $Mg(OH)_2$; however, the evidence for CSH II is enhanced in all mixtures, particularly by the 850 and 920 C exotherms. CSH I exothermic effects are also stronger in Figure 4 (9, 12). Some increase in intensity of the $Ca(OH)_2$ decomposition peak near 500 C is shown in Figure 1. The endothermic effects near 550 C in Figures 2 and 4 likely indicate the presence of a chlorite-like mineral (16, 18, 19, 20).

105 C for 12 Hours—DTA reactions at this temperature, shown in Figures 1 and 2, seem to be approximately the same as those at 40 C, so the same identifications are made.

145 C for 12 Hours—The DTA curves are almost identical for the monohydrated lime mixtures at 145 and 80 C, as shown in Figures 3 and 4 respectively. Those for the 145 and 80 C mixtures for dihydrated lime, shown in Figure 2, are also strikingly similar; therefore, identifications of constituents are the same as previously stated. The CSH I exotherm near 850 C is somewhat enhanced in the DTA of the monohydrated lime mixture as is the double exothermic effect for CSH II in the DTA of the dihydrated lime mixture (12).

The DTA curve shown in Figure 1 for the calcitic lime mixture may be characterized as predominantly CSH II with increased evidence for $Ca(OH)_2$ (3, 12).

156 C—The DTA curve shown in Figure 1 for the calcitic lime mixture is essentially that for CSH II with more evidence for $Ca(OH)_2$ than at lower temperatures (3, 12).

There is also more evidence in Figure 4 for the chlorite-like mineral and well-crystallized calcium silicate hydrate in the DTA curves for both types of dolomitic lime (6, 7, 9, 10). However, the nonexpanding 15.2 Å X-ray diffraction peak was present only for the mixture using monohydrated lime (16, 19, 20). Although there is diminished evidence for CSH II in the dihydrated lime mixture, other constituent identifications remain the same as for the 145 C mixture.

164 and 170 C for 12 Hours—As shown in Figure 1, DTA curves for these 2 calcitic lime mixtures reflect a transition from CSH II to a lime-rich phase, with excess $Ca(OH)_2$ present as well; these effects are followed by their conversion to 11.3Å tobermorite at 170 C (6, 7, 9, 10).

Tobermorite is predominant in DTA curves for both of the dolomitic lime mixtures as shown in Figures 2 and 4, with more conclusive evidence for the chlorite-like mineral in the monohydrated lime mixture. In addition, some unreacted $Mg(OH)_2$ is indicated in both mixtures.

Because $CaCO_3$ is believed to be present in relatively small quantities, the lower temperature endothermic effects noted near 785 C are attributed to this compound (1, 4, 10, 11).

The low temperature exotherm is unexplained in curve J in Figures 1, 2, and 4 for identical freshly prepared mixtures using all 3 types of limes.

Analysis Summary of Weight Loss Data

A complete set of weight loss curves was obtained only for the monohydrated lime mixtures reacted over a long period of 23 C and subsequently reacted hydrothermally at 40, 80, 145, 155, and 170 C. Examination of these data for this mixture, shown in Figures 3 and 4, provides summary findings as follows: The weight losses below 300 C for the mixtures reacted hydrothermally at 170 C were 20 percent higher than for the 23 C mixture. There was also a slight upward shift in the DTA peaks. Weight loss above 700 C for the 145 C mixture is double that for 23 C. The 170 C mixture has a weight loss equal to one and one-half that for 23 C. These low temperature and high temperature thermal effects may be related as follows: As indicated in X-ray analysis of the 23 C mixture, CSH gel, CSH I, $CaCO_3$, and C_4AH_{13}, as well as some unreacted lime and bentonite, were noted ([1]). The 145 C mixture was noted to contain predominantly CSH I, a chlorite-like mineral, some unreacted $Mg(OH)_2$, and $CaCO_3$. The 170 C mixture was predominantly 11.6Å tobermorite with some 15.2Å chlorite-like mineral, $CaCO_3$, and unreacted $Mg(OH)_2$. The evident influence of increase in temperature on the progression of the reaction may be summarized in the following way: Unreacted bentonite and $Ca(OH)_2$ in the 23 C mixture provided materials for production of CSH compounds at intermediate hydrothermal reaction temperatures. These in turn are utilized in part to form tobermorite as the reaction temperatures approach 170 C. Concurrent decomposition of C_4AH_{13} above 156 C provided additional Ca^{++} ions and Al^{+++} ions. Substitution of aluminum in the tobermorite lattice may also have occurred. Starting at 23 C, long-term reaction between Mg^{++} ions and bentonite probably produced the poorly crystalline chlorite-like mineral. In the monohydrated lime mixture, the degree of crystallinity of this 156 C product was sufficient to clearly indicate a nonexpandable 15.2Å X-ray diffraction peak; the 550 C DTA peak was also evident. The weight losses for the 550 C reactions were constant at all temperatures, and there was negligible change in either the $Mg(OH)_2$ peak or associated weight loss with increase in hydrothermal reaction temperature. Thus one may conclude that only a progressive refinement in degree of crystallinity of initial chlorite-like material occurred with increased hydrothermal reaction temperature.

Mixtures using both types of dolomitic lime reacted at 23 C showed DTA peaks and equal weight losses near 550 C. Although not conclusively supported by X-ray diffraction results obtained on the dihydrated lime, this effect may be attributed to formation of the poorly crystalline chlorite-like mineral. Weight loss for $Mg(OH)_2$ for the dihydrated lime mixture was 3 times that for the monohydrated lime. Losses near 140 and 700 C associated with CSH compounds were approximately the same for both types of lime. Carbonation of $Ca(OH)_2$ in the dihydrated lime mixture was apparently 3 times that in the monohydrated lime mixture from comparison of weight loss data obtained near 800 C.

Comparison of the weight loss data for calcitic lime mixtures reacted at 23 C with the dolomitic lime mixtures indicates about equal CSH product formation. The $CaCO_3$ formation is almost double that for the dihydrated lime mixture.

CONCLUSIONS

1. DTA, especially when accompanied by DTG and TG analysis, provides valuable data supplementary to that obtained by X-ray diffraction, as noted later.

2. DTA delineates between CSH I and CSH II, which may occur as mixture constituents having approximately equal degrees of crystallinity. Although the compounds may be indistinguishable by X-ray diffraction, the characteristic CSH II thermal effects are evident in a DTA pattern of the mixture.

3. Mixture constituents, having such low degrees of crystallinity that no X-ray diffraction peaks are obtainable, may be inferred from analysis of weight loss measurements obtained simultaneously with DTA curves. Detection of a bentonite-magnesium

alteration product in dolomitic lime-bentonite mixtures reacted at room-temperature for long periods is an example.

4. Hydrothermal reactions at elevated temperatures (105 to 170 C) for relatively short periods (12 hours) produce reaction products (e.g., tobermorite) having higher degrees of crystallinity than obtained at 23 C (e.g., CSH gel) after long-term reaction (3 years). Products (e.g., CSH I and CSH II), obtained at slightly increased temperatures (40 to 80 C) reacted for 4 to 5 days, are similar to those obtained at elevated temperatures (105 to 145 C) for relatively short periods of time (12 hours).

5. The higher temperature hydrothermal reactions (155 to 170 C) utilize constituents produced in lower temperature reactions. This may be inferred from analysis of weight loss measurements obtained simultaneously with the DTA curves. An example is the formation of tobermorite from available CSH I and II and other possible sources of calcium that have interacted with bentonite in the presence of water at 156 C and above.

6. The extent of carbonation of $Ca(OH)_2$ is clearly indicated from DTA and TG data to be greatest in the calcitic lime mixtures followed by those containing the dihydrated and monohydrated types of lime.

7. Very little difference is detectable in pozzolanic reaction at room temperatures between $Ca(OH)_2$ and bentonite in water for either the calcitic or dolomitic types of lime.

8. There was no detectable carbonation of magnesium compounds in any of the reactions studied.

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